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Pilot Plant Design for Chemical Desulfurization of Coal

TRW Systems Group, Redondo Beach, Calif

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PILOT PLANT DESIGN FOR CHEMICAL DESULFURIZATION OF COAL

by

L.J. Van Nice and M.J. Santy

TRW Systems Group
One Space Park
Redondo Beach, California 90278

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EPA Project Officer: L. Lorenzi

Industrial Environmental Research Laboratory
Office of Energy, Minerals, and Industry
Research Triangle Park, N.C. 27711

Prepared for

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ABSTRACT

This document presents the results of a program for design and operational planning of facilities for testing the Meyers Process for chemical removal of pyritic sulfur from coal. Two options were evaluated: (1) a complete pilot plant test of the process at a 1/2-ton per hr scale and (2) scale-up and testing of only the most critical portion of the process, the reactor and regenerator section (reactor testing unit).

The design and test planning effort for scale-up of the Meyers Process described herein includes: (1) a summary of background process data, (2) a discussion of the pilot plant design, (3) pilot plant start-up and operational test plans and (4) the preliminary design, start-up and test approach for the reactor testing unit. Eight appendices are included which contain the following: (1) process flow diagrams for the complete pilot plant, (2) pilot plant mass balance computer program, (3) pilot plant plot plans and a sketch of the facility, (4) complete pilot plant equipment list, (5) critical path schedule for construction of the pilot plant, (6) preliminary process flow diagrams for the reactor testing unit approach, (7) preliminary reactor test unit plot plans and a sketch of the facility, and (8) reactor test unit equipment list.

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METRIC CONVERSION FACTORS

British	Metric
1 Btu	252 calories
1 Btu	2.93×10^{-4} kilowatt-hours
1 kw	1,000 joules/sec
1 hp (electric)	746 joules/sec
1 psi	0.07 kilograms/cm^2
5/9 (°F-32)	°C
1 inch	2.54 centimeters
1 ft	0.3048 meter
1 ft^2	0.0929 meters^2
1 ft^3	0.0283 meters^3 or 28.3 liters
1 gallon	3.79 liters
1 pound	0.4536 kilograms
1 ton (short)	0.9072 metric tons

1. INTRODUCTION

This document presents the results of an Environmental Protection Agency sponsored program aimed at designing and planning the operation of test facilities for evaluation of the Meyers Process for coal desulfurization. A complete pilot plant test of the process is one option for the next step in the development of the Meyers Coal Desulfurization Process following the successful completion of essential bench-scale testing. A second option would involve scale-up and testing of only the most critical portion of the process, the reactor and regenerator section. The previous investigation[1] firmly established the technical and preliminary economic potential of the Meyers Coal Desulfurization Process and led to the definition of a baseline pyritic sulfur removal process which served as the basis for design and test planning of both a full pilot plant and a reactor testing unit.

A pilot plant design engineered under ground rules established by the EPA, outstanding in the breadth of its overall capability, was prepared. The plant is designed to remove 95% of the pyritic sulfur from raw run-of-mine high pyritic sulfur (>3 wt %) coal and to accomplish this with: (1) continuous automated operation much like a demonstration plant, (2) maximum flexibility of operation, and (3) detailed measurement of both the performance of all major equipment and the fate of all pollutant-forming constituents through extensive instrumentation. The plant design is sized to operate continuously for sustained periods of time at a coal throughput from 1/4 ton to 1/2 ton/hr. In addition, a secondary pilot plant reactor has been designed to do double duty as a tenfold geometric scale-up of the critical primary reactor/regenerator unit. Note that a further tenfold scale-up of the reactor/regenerator unit would be equivalent to the size which would be utilized in a 50 tph demonstration plant, with sufficient desulfurized coal output to feed a 100 to 150 MW power unit.

Alternate approaches to scale-up of the Meyers Process not requiring the complete and versatile pilot plant were also examined. This effort resulted in a preliminary design for testing the critical reactor/regenreactor section of the Meyers Process in much the same manner as in the pilot plant design. The less critical steps would be tested at equipment manufacturers to develop design and scale-up information for process steps downstream of the reactor section. The Reactor Test Unit design also includes the capability to evaluate the process for coarse coals (1/4 to 3/8 in. top size) which cannot readily be processed as coal slurry.

In addition to this summary report, the program output consists of: (1) a detailed construction bid book, suitable for obtaining fixed price bids on the construction of the pilot plant at the TRW Capistrano Test Site (San Clemente, California), (2) an estimate of the cost of constructing the facility at the TRW Capistrano Test Site, and (3) a scale model of the complete pilot plant.

The Construction Bid Book, which was submitted to EPA under separate cover, contains the following information:

- Scope of work to be performed during plant construction.
- Plot plans and general arrangement drawings of the facility.
- Piping and instrumental drawings.
- Selected mechanical equipment types, specifications and vendors.
- Piping specifications and line list.
- Insulation specification.
- Specification for foundation, sewers and paving.
- Structural specifications.
- Electrical specifications and drawings.
- Instrumentation summary and specifications.

- Insulation summary and specifications.
- Protective coatings specifications.

A construction cost estimate for the pilot plant, as specified in the Bid Book, has been prepared and submitted to EPA as a separate item. The cost estimate was based on written quotations from suppliers for all major equipment. Generally, quotations were received from three or more suppliers for each major item and the most advantageous or lowest cost source was selected for the estimate. Piping, valving and instrumentation costs were developed from catalog prices or recent procurement experience. Craft labor and construction materials were estimated using experience factors for similar projects.

A detailed scale model of the pilot plant (3/8 in. = 1 ft) was delivered to EPA. The model was constructed from the piping and instrumentation diagrams, the plot plans and the general arrangement drawings.

The following sections describe the design and test planning effort for scale-up of the Meyers Process for the chemical desulfurization of coal. Section 2.0 presents a summary of background information primarily obtained during bench scale testing. Section 3.0 discusses the pilot plant design. Pilot plant start-up (Section 4.0) and operational (Section 5.0) test plans are detailed. The preliminary design, start-up and testing of the reactor section rather than the complete pilot plant, is described in Section 6.0. This document also includes eight appendices which contain the following: (1) process flow diagrams for the complete pilot plant, (2) pilot plant mass balance computer program, (3) pilot plant plot plans and a sketch of the facility, (4) complete pilot plant equipment list, (5) the critical path schedule for construction of the pilot plant, (6) preliminary process flow diagrams for the reactor testing approach, (7) reactor test unit plot plans and a sketch of the facility, and (8) reactor test unit equipment list.

2. BACKGROUND

The Meyers Process is a developing method for the removal of pyritic sulfur from coal utilizing a regenerable aqueous ferric sulfate leaching system. An overall representation of the process chemistry for both leaching and regeneration is outlined below:

Process Description

(1) Crushed coal is treated with warm ferric sulfate solution

$$FeS_2 + 4.6Fe_2(SO_4)_3 + 4.8H_2O \rightarrow 10.2FeSO_4 + 4.8H_2SO_4 + 0.8S$$

(2) Generated sulfur is removed with a warm solvent bath or by vaporization

(3) Ferric sulfate solution is regenerated with air and excess ferric and ferrous sulfates are removed

$$9.6FeSO_4 + 4.8H_2SO_4 + 2.4O_2 \rightarrow 4.8Fe_2(SO_4)_3 + 4.8H_2O$$

The process operates under mild temperatures and pressure, i.e., 90 to 130°C and ambient to 100 psi or greater.

The coal feed for chemical desulfurization may range from finely ground coal (e.g., -100 mesh) to coarse coal suitable for shipping (e.g., -3/8 or -1/4 in.). The major emphasis in the development of the process to date has been on the processing of fine coal utilizing a simultaneous leaching and regeneration approach in which the ferric sulfate leaching agent is regenerated in the presence of the coal which is being leached.

Previous EPA-sponsored programs have been undertaken to (1) define the applicability of the process for reduction of sulfur oxide pollution from stationary sources through desulfurization of uncleaned run-of-mine

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coal, (2) prepare conceptual engineering designs of full-scale units, and (3) prepare cost estimates for commerical plants. These studies indicate that the Meyers Process is capable of removing 90 to 95% of the pyritic sulfur from most coals and that application of the process to run-of-mine Appalachian coal reserves would free high sulfur reserves comprising about 30% of the present Appalachian coal production for power production under EPA's New Source Performance Standards, while most of the remaining Appalachian coal can be desulfurized by the process to meet state SO_x standards. In addition, the process is applicable for meeting Federal and State standards for selected Eastern Interior, Western Interior and Western coal reserves. Engineering cost analyses indicate that the process will be cost-competitive with flue gas scrubbing and other coal conversion processes.

Very recently, emphasis has been placed on the processing of coarse coal. It has been found that more than 80% of the pyritic sulfur can be removed from coarse run-of-mine Appalachian coal in 48 hours, at 100°C, and that near complete removal of pyritic sulfur can be obtained on continued leaching. Further, the utilization of clean coal, typical of the output of a coal preparation plant, significantly increases the rate of pyrite removal by the Meyers Process.

Summary information on process data for desulfurization of both fine and coarse coal is presented in Section 2.1 and a summary of factors which can reduce commercial scale process costs is presented in Section 2.2.

2.1 PROCESS DATA

The majority of process design data on the Meyers Process were generated under two EPA sponsored bench-scale process evaluation and development programs (Contract EHSD 71-7 and Contract 68-02-1336).

Under Contract EHSD 71-7, data were generated which demonstrated the technical feasibility of all process unit operations. In addition, empirical rate expressions were derived for coal pyrite leaching (100 mesh x 0 Lower Kittanning coal) and reagent regeneration on which a preliminary process design and cost analysis was based. More recent data, presented in the following sections, cover results derived from simultaneous leaching-regeneration processing of suspendable coal (100 mesh x 0 and 14 mesh x 0 Lower Kittanning coal) and preliminary data on coarse coal (1/4 inch x 0). The suspendable coal experimentation was performed under EPA Contract 68-02-1336.

2.1.1 Simultaneous Coal Leaching-Reagent Regeneration

The primary objective of the second bench-scale program on the Meyers Process (EPA Contract 68-02-1336) was the investigation of potential process improvements aimed at process simplification and cost reduction. A number of the attempted process improvements proved successful; the most important were simultaneous leaching-regeneration, intermediate size coal processing (14 mesh x 0), and concentrated slurry processing (> 30 wt %).

In the combined leaching-regeneration mode of operation the coal to be processed was slurried in hot recycled reagent solution (nominally 5 wt % iron, 2 wt % H_2SO_4) in a mixing vessel, heated to boiling, and refluxed for 15 to 30 minutes prior to transfer to the leaching-regeneration pressure reactor (L-R reactor); this slurry-mixing, coal-wetting operation in the "mixer" minimized slurry foaming in the L-R reactor. The slurry was sampled at this point in order to determine the quantity of pyrite leached from coal during the slurry mixing-heating operation; the pyrite content of the coal at the end of this operation was considered the starting coal pyrite concentration for the L-R operation. After slurry transfer to the leaching/regeneration reactor, the system was pressurized by nitrogen to a nominal 100 psig and heated to the desired L-R processing temperature (nominally 120°C). As soon as the slurry temperature reached the desired value, slurry circulation (through a reactor loop) and oxygen injection to the slurry were initiated. L-R processing was varied from

1 to 6 hours in these experiments. Reactor slurry samples were also taken at frequent reaction time intervals (0.5 to 1.0 hours) during each experiment. Upon expiration of the desired L-R processing time, the reactor was depressurized and the slurry was either transferred to a settler for further coal leaching at 90 to 95°C or the leaching reaction was terminated by immediate filtration and cake washing. In either case, the leached coal was subsequently (1) washed with water to remove residual iron sulfate; (2) washed with toluene for recovery of elemental sulfur, (3) vacuum dried and (4) analyzed. The available data to date on L-R processing of suspendable Lower Kittanning coal leads to the following observations:

- Coal pyrite leaching rates under L-R processing at 120°C are approximately three times higher than those determined for 102°C coal leaching under continuous reagent exchange (separate reagent regeneration).

- Pyrite leaching rates derived from L-R processing at 120°C and 100 psig of 100 mesh x 0 and 14 mesh x 0 Lower Kittanning coal are virtually identical.

- Coal pyrite leaching rates at 120°C are not detectably affected when slurry concentration is increased from 20 wt % to 33 wt % solids.

- During the first 2 hours of L-R operation at 120°C and 100 psig (approximately 80% pyrite removal) the pyrite leaching rate from 100 mesh x 0 and 14 mesh x 0 Lower Kittanning coal can be represented by the empirical expression

  $r_L = k_L W_p^2 Y^2$ = wt pyrite removed/hr-100 wt coal where W_p = wt % pyrite in coal

Y = ferric ion to total iron wt ratio, and

k_L = 0.45 ± 0.05 (hours)$^{-1}$ (wt % pyrite)$^{-1}$

Pyrite leaching rates decrease substantially after approximately 2 hours of L-R operation; this observation may be characteristic of the particular coal under investigation and it could be due to the high ash content of the coal (30 wt %). Additional pyrite removal is attainable during settler processing (90 to 95°C, ambient pressure).

- L-R processing, at least up to 130°C and 100 psig, does not appear to affect the composition and heat content of the coal beyond those changes expected due to pyrite removal; however, changes in trace elements are still under investigation. Coal analysis of "as received" and processed coal furnished no evidence of either oxidation or sulfonation of the coal matrix.

- Reagent regeneration rates under L-R operation appear to be the same as those determined from separate regeneration operation. During 2 hours of L-R operation, the slurry Y value increases from approximately 0.5 to 0.9.

- 100 hours of reagent recycling under L-R conditions furnished no evidence of reagent efficiency deterioration.

The data generated on simultaneous coal leaching-reagent regeneration processing has not yet been completely reduced and evaluated; however the above conclusions on process performance appear valid. The formulated rate expression and the quoted value of its constant are considered adequate for scale-up designs for suspendable Lower Kittanning coal. The data listed below indicate typical pyritic sulfur (Sp) removals obtained during the processing of such coal (starting Sp = 4 wt %, starting ash 30 wt %):

	% Sp Removal
During slurry heating and mixing	15-20
After 1-hour L-R operation	~65
After 2-hour L-R operation	~80
After settler processing (20 hours at 90°C)	~90

2.1.2 Coarse Coal Data

Processing of coarse coal by the Meyers Process has not been as extensively investigated as the suspendable coal; however, the available data indicates that processing of coarse, readily shippable size coal is feasible and therefore merits further investigations.

Data on coarse coal were generated with 100 to 500 grams, 1/4 in. x 0 Lower Kittanning coal slurried in iron sulfate solution (5 wt % Fe, 15 to 20 wt % solids). The leaching temperature was maintained at 102°C (reflux under ambient pressure). The reaction time was varied from 4 to 48 hours. The reagent in the reacting slurry was exchanged frequently in order to maintain the Y value in the 0.8 to 0.9 range. The extent of pyrite removal as a function of reaction time was determined from ferrous ion production and from sulfur forms analyses of the processed and unprocessed coal. Pyrite removal at several representative times are as follows:

Reaction Time, hr	% Sp Removal
4	37
6	44
24	73
48	81

A sample of 1/4 in. x 0 Lower Kittanning coal was sieved into several narrow size fractions. The coal from several size fractions was processed for 48 hours as described above. The following results were obtained:

Size Fraction	% Sp Removal
4 x 8 Mesh	67
8 x 14 Mesh	83
14 x 28 Mesh	88
100 Mesh x 0	98
1/4 in. x 0 Starting Coal	81

It is evident that a rate expression for coal leaching must include a particle size relationship. Additional data will be necessary to obtain an improvement to the overall integrated rate expression which is currently in use.

2.1.3 Cleaned Coal Data

Recently, a sample of 8 mesh x 14 mesh Lower Kittanning coal was cleaned by float-sink and the 1.75 specific gravity float portion (80% w/w of the sample) was treated by the Meyers Process under the same 48 hour conditions described previously.

	% Sp Removal	Final % Ash	Final Btu
Uncleaned sample	83	15	13,260
Float sample	92	5	14,740

The results show that (1) clean coal reacts substantially faster than run-of-mine coal and (2) coarse coal (in this case, a cleaned narrow size fraction) can be desulfurized to near zero pyrite (Sp = .09 wt %).

2.2 COMMERCIAL SCALE PROCESS COSTS

Conceptual full-scale process designs with corresponding cost estimates have been prepared by TRW and independently by several engineering firms during several stages of the EPA sponsored laboratory and bench-scale development effort. Escalation of costs have occurred due to inflation since these data were generated and while the magnitudes have changed, it is probable that the ratio of cost items is relatively constant. Thus, in excess of one-half of the process cost of pyritic sulfur removal by the Meyers Process is related to the capital costs of the process. The areas where capital cost improvements are most likely are:

> Processing larger particle size coal: Generally the cost of separating the coal from leach solution will rapidly decrease as particle size increases. A decrease in separation cost during washing and solvent extraction will be partly offset by an increased contact time for solutions to penetrate and equilibrate with the internal pores of coarser coal particles.
>
> Processing cleaned coal: Three areas of cost reduction appear to be related to coal cleaning: (1) there is less pyrite to remove so that less residence time and oxygen are required, (2) reaction rates with cleaned coal appear to increase, further reducing residence time, and (3) filtration rates increase as the ash component of coal is decreased.
>
> Increasing slurry concentration: Increasing the coal concentration in the slurry will reduce the volume of the reactor, although not in direct proportion to the reduction in slurry volume. A major effect is that slurries with 33% and greater solids will not require a thickener and filtration cost decreases as solid content increases.

Alternate methods of removing elemental sulfur: Removal of elemental sulfur by extraction with an organic solvent has been evaluated in detail at bench-scale and is the basis for current process designs. Direct vaporization of sulfur using hot purge steam, or an inert gas such as nitrogen, has been evaluated briefly and has been found to be feasible and to be as effective as solvent extraction. Cost reduction is possible if the vaporization temperatures are low enough, residence time short enough and if few troublesome compounds are vaporized and condensed with the sulfur.

Updated process designs and process cost estimates will be prepared for commercial size plants as a final task in the current bench-scale program (EPA Contract 68-02-1336). These cost data and a preliminary assessment of potential benefits from gravity and size/gravity separation prior to leaching will be important in selecting the goals for further process improvement and cost reduction.

3. PILOT PLANT DESIGN

The process flowsheet and mechanical design of a pilot plant to evaluate the Meyers Process are complete. Ehrhart Division of Procon Incorporated, under a subcontract to TRW, provided the mechanical design and assisted TRW in the process design and preparation of the flowsheet.

Key ground rules in the design of the coal desulfurization pilot plant were established by the EPA in the Scope of Work of the Design Contract. Those were :

(1) The pilot plant shall be designed to have the capability of processing up to approximately one-half ton of coal feed per hour and shall be capable of processing the coal automatically in both batch and continuous flow modes.

(2) The pilot plant design effort shall be directed toward a flow scheme capable of total removal of pyritic sulfur from coal.

(3) The process design shall provide for sufficient instrumentation and sampling points as are necessary to obtain complete material and energy balances around the entire plant as well as around each major processing or material handling operation.

(4) The process design shall also allow for the determination of the fate of ash and other potentially pollutant-forming constituents in addition to determination of the fate of sulfur.

(5) Analytical techniques and equipment which can provide rapid, reliable and relevant process data shall be designed to provide for the collection of information on the performance of all major items of equipment and shall be designed to have sufficient flexibility to permit, with little difficulty, the timely incorporation of alternate processing units, as may be required from further process development, into the pilot plant.

(6) The pilot plant shall be designed to provide optimal economic supply of process heat requirements and shall provide an

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environmentally sound system and one which results in the most desirable utilization of fuel resources in recognition of the requirements for commercial application.

(7) Considerations during pilot plant design shall be devoted to the establishment of pilot plant control variables which are required for processing different types of coals.

The above plant design basis is almost unique in scope for pilot units in that (1) the plant will be an automated unit capable of continuous and batch operation, while (2) the plant will be capable of maximum flexibility of operation with provision for incorporation of alternate processing units. In addition, the plant will be thoroughly instrumented for extensive determination of both the performance of all major items of equipment and the fate of all pollutant-forming constituents.

The plant was designed to be constructed at TRW's 2700-acre Capistrano Experimental Facility on an existing four-level test stand which has ready access to power, cooling water, steam and potable water. The pilot plant plot plans and an artist's sketch of the facility are presented in Appendix C. The detailed design and specification of equipment, piping and instrutation diagrams, vendor specifications, etc., are included in the design package which is in a separate volume. Section 3.1 presents a description of the pilot plant process design (process flow diagrams are presented in Appendix A), Section 3.2 discusses the design basis (including the mass balances) and Section 3.3 discusses materials of construction.

3.1 PROCESS DESIGN

The process design consists of six major operations, namely (1) grinding and feeding coal to a mixer, (2) concurrent pyrite leaching and regeneration of the iron sulfate leach solution, (3) secondary leaching to reach the design level of pyrite removal, (4) water washing to remove leach solution and soluble sulfate from the coal, (5) toluene extraction to remove elemental sulfur from the coal, and (6) steam or nitrogen stripping of the coal to recover the solvent. These six main operations, the alternate processing modes and the supporting process equipment will be described in the following 12 subsections with references to stream numbers and equipment numbers shown on the flow sheet (Appendix A foldouts). A detailed pilot plant equipment list is presented in Appendix D.

3.1.1 Feeding and Grinding

Coal of about 1-1/2" top size is brought from storage by conveyor A-1 to the pulverizer system A-3. Pulverized coal is conveyed to surge tank T-1. The pulverizing system and surge tank are designed for operation under a nitrogen rich atmosphere and will vent excess gas through fabric filters for dust removal. The tank is equipped with a water spray system for fire protection.

3.1.2 Mixing

Ground coal is passed through the bin discharge A-4 to the weigh belt feeder A-5. The weighed coal is continuously fed through rotary feeder SP-8 to the first stage of the three-stage mixer T-2 (stream 1) where it is slurried with return leach liquid (stream 2) and heated to boiling by live steam (stream 3). Gas carried in with the pulverized coal and any gases (such as CO_2) liberated by reaction are washed by the incoming leach solution in SP-1 and with water in T-3 before being vented through blower B-2. The slurry and gases from any of the three stages can be sampled and analyzed.

3.1.3 Reaction and Regeneration

The heated slurry is pumped by P-1 to the reaction vessel R-1 (stream 4). The interior of the cylindrical reaction vessel is divided into ten compartments with a length approximately equal to slurry depth. Baffles are are provided to allow for cascade flow from stage to stage and to inhibit return of slurry to an upstream stage. The first five stages can be bypassed by the incoming slurry in order to operate as a five-stage reactor. Continuous regeneration of the leach solution in each compartment or stage is carried out by injection of oxygen (stream 5) into the discharge line of a slurry re-circulation pump connected to each stage (pumps P-2A through P-2J). Each stage is provided with an agitator with an impeller installed near the vapor-liquid interface in order to reduce the possibility of any significant build-up of solids in the vapor space.

In addition, two agitators are provided with a second impeller suitable for gas dispersion so as to demonstrate mechanical aeration as a means of leach solution regeneration. The two gas dispersion agitators can be installed in any compartment. A manifold system equipped with static mixers suitable for installation in a single stage is also provided for demonstration of a third means of leach solution regeneration. Injection connections for steam and for cool recycle leach liquor are provided for temperature control.

Steam, excess oxygen and inert gases are drawn from the reactor (stream 6), then washed and cooled by recycle leach solution (stream 17). Any water condensed from stream 6, water soluble components and mist are added to stream 17 to give the mixer feed (stream 2). The washed, cooled gas is split. A small portion (stream 9) is vented through scrubber T-3 while the major portion (stream 8) is recycled to the reactor through compressor K-1. Makeup oxygen is provided by stream 7.

3.1.4 Secondary Reactor

Slurry from reactor R-1, under pressure (stream 10), is reduced to about atmospheric pressure and introduced to a flash drum (T-4). Flash steam (stream 11) is condensed with cooling water. The remaining slurry, at its boiling point (stream 12), is introduced to the secondary reactor (R-2). This two-stage vessel is similar to R-1 but each stage is about an order of magnitude larger in volume. Alternate piping is provided so that the secondary reactor can also be operated as a two-stage primary reactor where the two stages are geometrically about ten times bigger than the stages of reactor R-1. Using R-2 in this fashion will provide data on the geometric scalability of the reactor system.

3.1.5 Slurry Concentration

The effluent from reactor R-2 (stream 13) is pumped (P-3) to the first filter (S-2). Slurry concentration may be as low as 10% solids in stream 13. Significant saving in filtration cost can be realized if preconcentration of the slurry into the 30 to 50% solids range can be achieved. Two off-line processes with potential for slurry concentration are shown.

A demonstration thickener (R-3) is provided to intermittently test a portion of the secondary reactor effluent to determine the extent that slurry thickening can be achieved and whether a thickener can function as a secondary reactor. Design is based on manufacturer's recommendations for a pilot thickener operation. Underflow is pumped (P-4) to the filter (S-2) and the overflow (if it is sufficiently clear) will go to surge (T-9). A further task of the thickener operation is to test acid resistant concrete lining of steel equipment and to test rubber coated thickener internals.

Piping and flow controls are provided for testing the slurry thickening achieved in a hydroclone. Several sizes from 1/2 in. to 3 in. are provided to examine the separation achieved for coal slurries varying in particle size from 100% -8 mesh to 100% -100 mesh. This system generally will be operated off-line with slurry from R-2 pumped (P-26) through the hydroclones to mixed surge tanks for the underflow (T-26) and overflow (T-27). Provisions are also made to operate two hydroclones in series so as to reduce the solids content of the overflow.

3.1.6 First Filtration and Washing

The nominal operating mode of the plant involves filtering the slurry from reactor R-2 (stream 13) to provide recycle leach solution and then further washing the cake to reduce the residual sulfate on the coal. Filter S-2 is a fully enclosed rotary vacuum filter. The filtrate (stream 19) from the initial separation of the feed slurry is pumped (P-8) from the vacuum receiver (V-2) to the recycle surge tanks (T-9 and T-10). The filter cake is spray-washed in the filter with a dilute leach solution (stream 22) and the wash liquid is drawn to the second vacuum filtrate receiver (V-3). Pump P-9 draws liquid from V-3 (stream 20) and sends a portion (stream 18) to the disposal tank (T-25) and a portion (stream 15) to the makeup tanks (T-6 and T-7). Any makeup materials are added to T-6 and T-7 and the resultant stream 16 is pumped (P-5) to the surge tanks (T-9 and T-10). The combined filtrates and makeup contained in tanks T-9 and T-10 are pumped to the reactor knock out drum V-1 as stream 17, which was previously discussed. Vacuum for the filtrate receivers (V-2 and V-3) is provided by compressor K-2 operating through a barometric condenser (E-1).

3.1.7 Crystallizer System

It should be noted that the pilot plant process normally disposes of the iron sulfate from the pyrite leach reaction as an aqueous solution (stream 18 principally). The commercial design plans to recover wash water from the waste stream and to produce a solid iron sulfate product. The crystallizer system (SP-3) is designed to investigate this aspect of the processing. A separate package system is provided to test the effect of temperature and feed composition on the iron/sulfate ratio and the water of hydration obtained in the crystal product. The crystallizer-evaporator will operate intermittently on a portion of the leach liquor drawn from surge tanks T-9 and T-10. The unit is designed to evaporate 1000 lb per hour of water operating at atmospheric pressure or under vacuum. The product crystals will be separated from the rich liquor using the first centrifuge in the organic extraction section (S-6). Filtration tests may also be conducted with filter S-2 without the spray-wash.

3.1.8 Final Washing

The filter cake from the first filter (stream 21) is given additional washing to reduce the soluble sulfate level. The steps include two separate stages of repulping, filtration and filter cake washing. The first repulping occurs in contactor T-11 where the filter cake (stream 21) is slurried with recycled wash water (strwam 23) and then pumped (P-10) to the second filter (S-3). The second filter is operated much like the first filter with a wash stream (27) and two filtrate streams. The receiver effluents from the vacuum receivers V-4 and V-5 are separately pumped (P-11 and P-12) to the common header (stream 54) which is sent to waste disposal tanks (T-25A and B). Vacuum for the receiver is provided by K-3 through barometric condenser E-2. The filter cake (28) is repulped with water (29) in a mix tank (T-13) and pumped (P-13) to the last filter (S-4). In this filter, the filtrate from the feed (stream 30) and from the water wash (stream 31) are combined in the vacuum receiver (V-6). The combined filtrate and wash (stream 32) is pumped (P-14) to the surge tank (T-20). This very dilute solution (stream 53) is pumped (P-19) back to the first washing stage to provide the wash to the first filter (stream 22) and the water for the first repulping mixer (stream 23). Vacuum for the final filter (S-4) is provided by K-4 and condenser E-3.

3.1.9 Elemental Sulfur Removal

Removal of elemental sulfur from the coal is accomplished by extraction with hot toluene. Water-wet coal from the final wash filter (stream 33) is introduced to a jacketed vessel (T-14) through a rotary feed valvn (SP-7). Sulfur lean toluene (stream 34) is added to T-14 and the water/toluene azeotrope is removed (struam 35), condensed (E-6) and fed to decanter T-19 for phase separation. The water-free coal toluene slurry (stream 36) from T-14 is pumped (P-15) to the first centrifuge (S-5). Clean toluene (stream 38) is used to wash the cake and a combined centrate/wash solution (stream 37) is pumped (P-16) from the receiver (T-15) to the rich solvent surge tank (T-22). The cake from S-5 (stream 39) is repulped in T-16 with fresh toluene (stream 40) and the resultant slurry (stream 41) is pumped (P-17) to the second centrifuge (S-6). The cake is washed with additional toluene (stream 43) and the combined centrate wash (stream 42) is transferred to surge tank T-21.

3.1.10 Coal Stripping

Coal, wet with toluene from the second centrifuge (stream 44), is fed through a rotary feeder (SP-6) to the continuous rotary shelf dryer (SP-4). Hot nitrogen (or steam) is used to strip the toluene from the coal to produce a dry coal (stream 47) which is cooled to give the product coal. When steam stripping is used, the input steam (stream 46) is removed from the dryer (stream 45) with toluene from the coal. Stream 45 is condensed (E-5) and separated in T-18 to give toluene for reuse (stream 49) and water for disposal (stream 50). With a hot nitrogen loop, decanting is not required; but a knockout drum would be used to separate the toluene from the recycle nitrogen.

3.1.11 Sulfur Recovery

Toluene, rich in sulfur, from the first centrifuge is removed from the surge tank T-22 by pump P-21 and fed (stream 56) to the solvent still (C-1). Clean toluene from the still overhead (stream 58) is condensed (E-7) and sent to T-23 where it is combined with clean toluene from the azeotropic still decanter (stream 51) and from the dryer decanter (stream 49). The clean, heated toluene (stream 59) from T-23 is pumped (P-22) to the centrifuges (streams 38 and 43), to the second repulper (stream 40), and to T-21 (stream 55) where it is combined with another lean sulfur stream (42). The lean sulfur stream from T-21 (stream 34) is pumped (P-20) to the azeotropic still as discussed in Section 3.1.9.

Returning to the solvent still (C-1), the sulfur rich bottoms (stream 60) are pumped (P-23) to the scraped wall exchanger (E-9). The sulfur by-product (stream 61) is removed from the toluene in either of two leaf filters (S-7 and S-8). In addition, a portion of the stream from tank T-22 is bypassed around the still (stream 64) and fed to the filter to hold the sulfur cake on the filter leaves. The combined filtrate (stream 57) is returned to the surge tank (T-22).

3.1.12 Disposal

Disposal tank T-24 contains water from the azeotrope decanter (stream 52), and, when steam stripping drying is used, water from the dryer decanter (stream 50). Since there may be traces of toluene mixed with the water, this stream (63) is intermittently pumped (P-24) to the disposal truck. Commercial disposal is also used for tanks 25A and B, which contain the waste iron sulfate product from the pyrite reactor. The effluent (stream 62) is also intermittently pumped (P-25) to a disposal truck. A vapor absorber (SP-5) is used to control any vapor effluents. The carbon drum has been designed to hold approximately 500 pounds of carbon and this has been calculated to be adequate for 16 weeks of operation, assuming one complete vapor displacement of all solvent-containing tanks once per week.

3.2 DESIGN BASIS

The pilot plant mechanical design required nominal values of many parameters to be specified and, in some cases, also required minimum or maximum values to be known. The principal process parameters which influence flowrates and equipment selection or sizing are identified in Section 3.2.1. The nominal mass balance and the influence of parametric variations are given in Section 3.2.2. Additional equipment design information if given in Section 3.2.3.

3.2.1 Process Parameters

3.2.1.1 Mixer/Reactor Section - Stage-by-stage calculations of the extent of reaction in the mixer and in the two reactors were completed based on data from the bench-scale program as given in the final report[1]. It was calculated that 17.4% pyrite removal would occur in a mixer with three stages each of 0.25 hour residence time at about 100°C. After ten 0.5 hour reactor regenerated stages at about 120°C, the reaction was calculated to be 53.6% complete. The target 95% removal could be obtained with about 24 hours of additional reaction at 100°C. These values were taken as normal reaction extents. The ferric to total iron ratio (Y) in the recycled leach solution is dependent upon the amount of concurrent reaction and regeneration. At a reactor total pressure of about 50 psig, the above pyrite removals are obtained at a recycle Y of about 0.75 with nominal leach solution. Improved estimates of these rates will be obtained (from a current bench-scale program recently completed) and some adjustments to residence time and pressure may be necessary to obtain these precise removals and Y values. The nominal values of parameters necessary to provide a mass balance around the mixer/reactor section are as follows:

Coal feed rate (dry)	1000 lb/hr
Coal moisture	10 %
Pyritic sulfur (dry basis)	3.2 %
Iron in recycle leach solution	5.0 %
Solids content in mixer	20 wt.%
Steam/condensate to heat mixer	274 lb/hr
Reaction in mixer	17.4 %
Pressure in reactor R-1	50 psig
Temperature in R-1	250 °F
Water vapor pressure in R-1	29.8 psi
Oxygen concentration in R-1 exit	90 wt.% (dry)
Oxygen in makeup	99.5 wt.%
Water vapor pressure in vent (T=150°F)	3.7 psi
Oxygen fed to R-1/oxygen consumed	8.0
Flash steam from R-1 (stream 11)	150 lb/hr
Reaction in R-1 (83.6-17.4)	66.2 %
Reaction in R-2 (95.0-83.6)	11.4 %
SO_4/Fe ratio in R-2 effluent	1.5
Y in R-2 effluent	.75

It should be noted that the basic overall reaction for leaching pyrite and regenerating the solution is:

$$FeS_2 + 2.4\ O_2 \rightarrow 0.6\ Fe^{+2} + 0.4\ Fe^{+3} + 1.2\ SO_4^{=} + 0.8S$$

It can be seen that the inherent Y of this reaction is 0.4 and if the iron and sulfate are not removed in the ratios produced, the recycle stream will change composition with time. The pilot plant process proposes to remove the reaction products by non-selectively discarding a portion of the reactor (R-2) effluent. This would produce an imbalanced iron/sulfate ratio and Y in the recycle stream, if makeup is not correctly chosen. To keep the stream in balance the basic reaction also must be regenerated to equal the recycled Y (0.75 in the nominal design). The overall reaction is:

$$FeS_2 + 2.4875\ O_2 + 0.175\ H_2SO_4 \rightarrow 0.25\ Fe^{+2} + 0.75\ Fe^{+3}$$

$$+ 1.375\ SO_4^{-2} + 0.175\ H_2O + 0.8S$$

It is evident that sulfuric acid is consumed when the removal of iron forms is non-selective. The baseline design actually adds enough sulfuric acid that regeneration to a Y = 1.0 could be tested and results in additional sulfuric acid makeup and discard as follows:

$$FeS_2 + 2.4875\ O_2 + 0.3\ H_2SO_4 \rightarrow 0.25\ Fe^{+2} + 0.75\ Fe^{+3} + 1.375\ SO_4$$

$$+ 0.125\ H_2SO_4 + 0.175\ H_2O + 0.8S$$

3.2.1.2 Washing Section - The major parameters affecting the washing section mass balance are the moisture content of filter cakes and the effectiveness of spray-wash and repulping. Based on vendor tests and bench-scale filtration experience with Lower Kittanning coal of 100 mesh top size, the following design basis was formulated. This coal (which is believed to be typical of Appalachian bituminous coals) produces a filter cake which has a liquid retention equal to 50% of the dry coal weight plus the weight of dissolved salts. For example, coal filtered from water has 50% liquid but coal filtered from the 5% iron leach solution has 63.6% liquid because the liquid contains 21.4% of dissolved iron sulfates and acid.

Cake washing appears to be best represented by considering the retained liquid to be of two types. "Pore" liquid is about 15% of the coal weight plus dissolved salts. Spray-washing on the filter, with water equal to 1.4 times the water content of the cake, appears to replace substantially all of the "surface" liquid with wash liquid but does not change the "pore" liquid. The repulping step has enough residence time that both the surface and pore liquids equilibrate with the bulk liquid.

The nominal values of parameters used in the wash section mass balances are:

Filter cake pore water	15% of solids
Filter cake surface water	35% of solids
Filter spray-wash/cake water	1.4
Repulped slurry concentration	33% solids

5.2.1.3 Toluene Section - This section includes azeotropic drying to remove water, toluene washing to remove sulfur, and solvent recovery. Water-wet filter cake is slurried with boiling toluene and an azeotrope of water and toluene is removed. The azeotrope theoretically contains 55.6 mole percent water. But, because of imperfect contact, it was assumed that 5% excess toluene would be present in the azeotrope.

The toluene-coal slurries are separated by centrifuges which operate effectively with the large density difference between coal and toluene. A division of the centrifuge cake liquid into "pore" toluene and "surface" toluene was made. The pore toluene is assumed to be 13% of the dry cake weight plus dissolved sulfur. The 13% is slightly less than 15% water owing to the lower density of toluene. The total liquid was found by vendor test and bench-scale experience to be about 33% which results in a nominal "surface" toluene of 20%. Considering the lower density, viscosity and surface tension of toluene compared to water, this value is reasonable. Spray-washing in a centrifuge is less efficient than on a filter and it was assumed that two (rather than 1.4) times the cake toluene will be needed to displace the "surface" liquid.

The sulfur-rich toluene solution is purified by distillation (C-1). The still feed containing about 2% sulfur is concentrated to 10% sulfur, then cooled in a scraped tube exchanger to give a slurry of sulfur in sulfur-saturated toluene. The saturated stream will have nominally 2.7% dissolved sulfur at 95°F. At pilot scale, the flow rate of this stream is so low that a bypass stream (64) has been added to the filter feed to hold the sulfur cake to the leaves of the filter. Based on vendor experience, the liquid flow through the sulfur cake should be about 0.15 gallons per minute per square foot of filter area.

Toluene is also recovered from the coal in the dryer. In the nominal case, it was assumed that no toluene is lost with the coal. In practice, there will be a small loss and a corresponding amount of makeup will be required. If 0.1% of the coal weight is assumed to be absorbed toluene, then about 0.94 lb/hr of makeup is required.

Nominal values of parameters used in the toluene section mass balance are the following:

Centrifuge cake pore toluene	13% of solids
Centrifuge cake surface toluene	20% of solids
Centrifuge spray-wash/cake toluene	2.0
Repulped slurry concentration	33% of solids
Sulfur in still bottoms	10 wt.%
Sulfur solubility of filtrate	2.7 wt.%
Filtrate flow rate	0.15 gpm/ft²
Azeotrope-actual toluene/ theoretical	1.05
Steam for stripping toluene in dryer	260 lb/hr

3.2.2. Mass Balance

The nominal mass balance for the pilot plant is given in Table 1. The flow rates are given for each of the 64 streams as numbered on the flowsheet in Appendix A. Table 2 correlates all feed and product streams to their composition and service. Stream 47 shows that the coal product

TABLE 1. PILOT PLANT MASS BALANCE

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STREAM NO.	1	2	3	4	5	6	7	8
			[illegible]					
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
			[illegible]					
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOTAL WT.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

TABLE 7 (Continued). PILOT PLANT MASS BALANCE

[illegible]

TABLE 1 (Continued). PILOT PLANT MASS BALANCE

STREAM NO.	17	18	19	20	21	22	23	24
				FLOW RATE, LB-MOLE/HR				
COAL	0.0000	0.0000	0.0000	0.0000	[illegible]	[illegible]	0.0000	[illegible]
PYRITE	0.0000	0.0000	0.0000	0.0000	[illegible]	0.0000	0.0000	[illegible]
SULFUR	0.0000	0.0000	0.0000	0.0000	[illegible]	[illegible]	0.0000	[illegible]
FESO4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
FE2(SO4)3	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
H2SO4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
WATER	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
OXYGEN	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
INERT GAS	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
				FLOW RATE, LB/HR				
COAL	0.00	0.00	0.00	0.00	[illegible]	0.00	0.00	[illegible]
PYRITE	0.00	0.00	0.00	0.00	[illegible]	0.00	0.00	[illegible]
SULFUR	0.00	0.00	0.00	0.00	[illegible]	0.00	0.00	[illegible]
FESO4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
FE2(SO4)3	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
H2SO4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
WATER	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
OXYGEN	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
INERT GAS	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL WT.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

TABLE 1 (Continued). PILOT PLANT MASS BALANCE

PAGE 4 OF 5

STREAM NO.	25	26	27	28	29	30	31	32
				FLOW RATE, LB MOLE/HR				
COAL	0.0000	0.0000	0.0000	72.2179	0.0000	72.2179	0.0000	0.0000
PYRITE	0.0000	0.0000	0.0000	[illegible]	0.0000	[illegible]	0.0000	0.0000
SULFUR	0.0000	0.0000	0.0000	[illegible]	0.0000	[illegible]	0.0000	0.0000
FESO4	[illegible]	[illegible]	0.0000	[illegible]	0.0000	[illegible]	0.0000	[illegible]
FE2(SO4)3	[illegible]	[illegible]	0.0000	[illegible]	0.0000	[illegible]	0.0000	[illegible]
H2SO4	[illegible]	[illegible]	0.0000	[illegible]	0.0000	[illegible]	0.0000	[illegible]
WATER	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
OXYGEN	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
INERT GAS	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
				FLOW RATE, LB/HR				
COAL	0.00	0.00	0.00	[illegible]	0.00	[illegible]	0.00	0.00
PYRITE	0.00	0.00	0.00	[illegible]	0.00	[illegible]	0.00	0.00
SULFUR	0.00	0.00	0.00	[illegible]	0.00	[illegible]	0.00	0.00
FESO4	[illegible]	[illegible]	0.00	[illegible]	0.00	[illegible]	0.00	[illegible]
FE2(SO4)3	[illegible]	[illegible]	0.00	[illegible]	0.00	[illegible]	0.00	[illegible]
H2SO4	[illegible]	[illegible]	0.00	[illegible]	0.00	[illegible]	0.00	[illegible]
WATER	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
OXYGEN	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
INERT GAS	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL WT.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

TABLE 1 (Continued). PILOT PLANT MASS BALANCE

STREAM NO.	33	34	35	36	37	38	39	40
FLOW RATE, LB-MOL/HR								
COAL	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
PYRITE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
SULFUR	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
WATER	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
INERT GAS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
FLOW RATE, LB/HR								
COAL	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
PYRITE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
SULFUR	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
WATER	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
INERT GAS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOTAL WT.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

TABLE 1 (Continued). PILOT PLANT MASS BALANCE

PAGE 6 OF 8

STREAM NO.	41	42	43	44	45	46	47	48
			FLOW RATE, LB-MOLE/HR					
COAL	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
PYRITE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
SULFUR	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
FECL3	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
FECL2.4H2O	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
WATER	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
OXYGEN	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
INERT GAS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
			FLOW RATE, LB/HR					
COAL	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
PYRITE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
SULFUR	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
FECL3	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
FECL2.4H2O	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
WATER	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
OXYGEN	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
INERT GAS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOTAL WT.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

TABLE 1 (Continued). PILOT PLANT MASS BALANCE

[illegible]

STREAM NO.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
				FLOW RATES, LB-MOL/HR				
COAL	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
WATER	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
INERT GAS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
				FLOW RATES, LB/HR				
COAL	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
WATER	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
INERT GAS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOTAL WT.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

TABLE 1 (Continued). PILOT PLANT MASS BALANCE

[illegible]

STREAM NO.	57	58	59	60	61	62	63	64
				FLOW RATE, LB-MOL/HR				
COAL	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
INERT GAS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
				FLOW RATE, LB/HR				
COAL	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOLUENE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
INERT GAS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOTAL WT.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

TABLE 2. FEED AND PRODUCT PROCESS STREAMS

	Process Feed Streams					Process Product Streams					
Stream No.	1	2	7	16	46	11	13	47	61	62	63
Service	Coal Feed	Steam to Mix Tank	Oxygen Feed	Leach Soln. Makeup	Wash Water Feed	Flashed Steam	Leach Soln. to Disposal	Coal Product	Sulfur Product	Waste to Disposal	Waste to Disposal
Composition Tph											
Coal	940.13							940.13			
Pyrite	58.87							2.99			
Sulfur								0.70	11.90		
$FeSO_4$							13.07	0.38		17.56	
$Fe_2(SO_4)_3$							42.74	0.77		70.91	
H_2SO_4				72.95			3.46	0.07		5.80	
Water	700.00	374.00		-0.16	2,270.33	700.00	413.30			2,274.97	477.64
Toluene											
Oxygen			38.84								
Inert Gas			0.88								
Total	1,700.00	374.00	39.72	72.80	2,270.33	700.00	469.81	942.54	11.90	2,409.84	477.64

(944 lb/hr) at 95% pyrite removal will contain the following sulfur forms:

Pyrite sulfur	0.17%
Sulfate sulfur	0.01%
Elemental sulfur	0.02%

The mass balance also shows that the makeup stream (14) contains 14 lb/hr of sulfuric acid and -3 lb/hr of water. The negative water means water will be removed from the recycle loop. In practice, significant quantities of water will be evaporated into the vacuum systems during filtration. The water removed from the recycle stream by the first barometric condenser was not included in the mass balance, but, from a separate estimate, it is likely that about 200 lb/hr of makeup water will be needed.

The nominal mass balance given in Table 1 is the output from a computer model of the process written in Fortran for a time-sharing computer. The program is presented in Appendix B. The program also was used to examine the effect of varying selected parameters on the mass balance. The resultant calculated mass balances indicated that the largest differences in residual sulfate levels in the product coal were obtained in runs where the "pore" water on the filter cakes was increased from the 15% nominal to 25%. The sulfate sulfur increased from 0.01% to 0.03%. Similarly, increasing the "pore" toluene from the nominal 13% to 25% increased the elemental sulfur from 0.02% to 0.05%.

Other changes influenced stream sizes and compositions, but had little influence on the coal product. It was found that, as the percent iron in the leach solution is decreased from 5% to 2%, stream 20 contains insufficient iron to dispose of the reaction product, thus requiring that a portion of stream 19 be bled off for disposal. It was concluded that the sizing and arrangement of the pilot plant equipment and piping should readily accommodate the anticipated variable studies which are discussed in the test plan.

3.2.3 Additional Equipment Criteria

For some of the major items of equipment a brief description of additional design parameters are included in the following paragraphs:

Coal Pulverizer System (A-3): the design rate for the coal pulverizer is 1000 lb/hr. Feed size is 1-1/2 in. The unit is designed to produce material 100% minus any required standard screen size from 8 mesh to 100 mesh by changing the screens in the sieve unit or by changing conditions in the pneumatic classifier. The unit is designed for operating under a nitrogen rich atmosphere and will vent excess purge gas through fabric filters for dust removal.

Ground Coal Storage Tank (T-1): the ground coal storage tank is designed for storage of 2-1/2 day supply of coal based on a consumption rate of 1000 lb/hr.

Mix Tank (T-2): the slurry mixer is designed to provide 15 minutes residence time in each of three agitated stages so as to remove any foam formed due to release of carbon dioxide from reaction of carbonate minerals with the acidic leach solution.

The mixer is circular in cross section and operates 80% full with the length of each compartment approximately 80% of the diameter. The slurry cascades from stage to stage through an adjustable baffle (to reduce residence time) so as to provide complete mixing in any one stage but prevent return of the slurry to an upstream stage. Any gas evolved is evacuated with a blower after first scrubbing with incoming leach liquor to remove foam and finally scrubbing with plant cooling water to remove traces of entrained material from the first scrubber and any visible water vapor. Any possible entrainment is estimated to add less than 1% of additional dissolved solids to the normal level of approximately 1230 PPM total dissolved solids already present in the plant cooling water.

Primary Reactor (R-1): the primary reactor is designed for a hold-up of 5 hours operating 80 to 90% full. Design pressure and temperature are 120 psig and 275°F with nominal operating conditions of 60 psig and 250°F.

Secondary Reactor (R-2): the secondary reactor is designed for a residence time of 24 hours operating 100% full. The purpose of the secondary reactor is to complete the conversion of the pyrites in the presence of leach liquor which has reached a relatively high level of regeneration in the primary reactor. The secondary reactor is baffled to reduce back mixing and to promote plug flow through the reactor.

Oxygen and Slurry Recirculation (P2A to J and K-1): recirculated oxygen is introduced into the discharge line of a recirculating pump connected to each compartment of the primary reactor. The pumps are designed to recirculate the contents of a reactor stage about once every 9 minutes. Sufficient oxygen recirculation capacity is provided to feed a volume of gas up to 1/2 the liquid flow.

The oxygen is recycled and the inerts allowed to concentrate from 0.5% in the makeup oxygen (99.5% oxygen) to about 10% inerts in the recirculating gas (90% oxygen, dry gas basis). The inerts are then bled to atmosphere after scrubbing to remove steam and any traces of entrained solids.

Filters (S-2, S-3, S-4): the filter section is comprised of three types of filters. The size of each filter, based on laboratory filtration tests, is 40 sq.ft.

Slightly different types were selected so that a comparison could be obtained on almost identical slurries for future full-scale plant filter selection. Types of filters are as follows:

(1) St[illegible]ard rotary drum

(2) De[illegible] filter (to determine if back washing the filter cloth to prevent blinding is useful)

(3) High speed rotary drum with blow-back cake discharge

Two of the filters will be equipped with separate filtrate and wash water separators. All the filters will be equipped with wash water headers and cake steaming headers. All the filters are equipped with separate vacuum systems designed to handle four SCFM/sq.ft of filter area at vacuum of 24 in. of mercury.

Wash Water Contactors (T-11, T-13): the filter cakes from the first two filters are repulped with hot water in agitated vessels. The purpose of the vessels is to provide adequate time for extraction of the soluble sulfates from the pores of the coal. A residence time of 30 minutes is provided.

Azeotrope Still (T-14): design of the azeotrope still is based on sufficient hold-up time to allow for displacement of absorbed water with toluene, and for adequate heat transfer surface. The basis for design is a heat flux of 15,000 btu/hr/sq.ft. This fixes a jacketed vessel size of about 500 gals.and a residence time of about 80 minutes.

Centrifuges (S-5, S-6): coal is separated from the azeotrope still and solvent contactor slurries using solid bowl centrifuges. The two units specified are 12 in. diameter by 30 in. bowl equipped for washing the cake.

Solvent Contactor (T-16): the solvent contactor is designed for a 30-minute residence time.

Solvent Still (C-1): the purpose of the solvent still is to concentrate rich toluene containing about 2% sulfur up to 10% sulfur in the still effluent. The still is a 200-gallon jacketed vessel sized to provide the required duty at a flux of 10,000 btu/hr/sq. ft.

Sulfur Removal (E-9, S-7, S-8): the toluene-sulfur solution from the still is cooled to 95°F in a scraped surface cooler and the discharge containing the solidified or crystallized sulfur is pumped through a leaf-type filter and the toluene filtrate returned to the rich solvent surge tank. The filter is periodically drained, blown dry with nitrogen and the sulfur cake discharged to a commercial waste disposal bin located underneath.

Dryer and Solvent Removal System (SP-4): the toluene wet coal from the final centrifuge is introduced through a screw conveyor into a rotary tray type dryer where the solids are contacted countercurrently with heated nitrogen (or steam). The gas-toluene mixture from the top of the dryer is condensed and the toluene is returned to the process.

The coal from the exit of the dryer is cooled in a screw-type cooler and then elevated to the top operating level for loading the coal into drums and removal to the storage area.

Leach Products (T-24, T-25A and B): ferrous and ferric sulfate formed from pyrite during the leach reaction are collected in waste disposal tanks along with dilute wash water which contains small quantities of iron sulfates. Two 20,000-gal. polyester-fiberglass tanks are provided for collection of the waste material from six days of continuous operation. A separate stainless steel waste tank is provided to dispose of water containing traces of dissolved toluene. Both wastes will periodically be collected by commercial waste disposal firms.

3.3 MATERIALS OF CONSTRUCTION

Selection of materials of construction for the various pilot plant equipment and hardware was based on (1) knowledge of the state of the art of materials applications and (2) materials compatibility studies carried out by TRW as part of EPA Contract No. 68-02-1336. The construction materials selected for utilization in all sections of the pilot plant facility except those in hot leach solution-coal slurry service were determined through application of materials technology knowledge with verification from equipment and hardware suppliers. The materials compatibility studies were performed to evaluate a number of materials in hot leach solution-coal slurry service. This testing was necessary because of an extreme lack of available information in regard to corrosion and/or erosion characteristics of construction materials in contact with ferric-ferrous sulfate solution-coal slurries in the presence of gaseous oxygen (as in the primary reactor and circulation loops) at elevated pressures and temperatures. Results of the experimentation indicated that 316L stainless steel is the preferred material under reactor simulated conditions. Type 304/304L stainless, a more common and less costly alloy, gave variable results. The higher risk of failure did not justify its use. Armco 22-13-5 and Carpenter 20 Cb-3 appear to be more resistant than 316L under test conditions. Although these alloys are normally more costly, either one could be substituted for 316L in a situation when it offers a cost advantage or if required for meeting delivery schedule.

The specific materials selected for utilization throughout the facility are detailed in the Pilot Plant Bid Book in piping and instrumentation drawings and equipment specifications. However, the following summary does present the general philosophy of materials selection as applied to the pilot plant.

- Mechanical equipment in acid (leach solution) service - 316L stainless steel
- Mechanical equipment in organic solvent service - carbon steel
- Mechanical equipment in sulfur service - carbon steel
- Vessels and tanks in dilute acid, water, aqueous waste, or ambient leach solution service - fiberglass reinforced polyester
- Vessels and tanks in organic solution (including dilute organic waste solutions) service - carbon steel
- Vessels and tanks in hot leach solution service - 316L stainless steel

- Piping in leach solution (concentrated and dilute) service - 316L stainless steel
- Piping in oxygen service - 316L stainless steel
- Piping other than that in leach solution and oxygen service - carbon steel.

4. PILOT PLANT START-UP AND OPERATIONAL VERIFICATION

The process of initiating pilot plant operation will include personnel familiarization with the facility, initial start-up and shut-down for the purpose of functionally testing all associated process equipment and techniques during plant operation, and the performance of an operational verification test run. Necessary modifications to the operational procedure and to the plant itself will be made during the start-up period. It is anticipated that 3 months will be required to carry out the systematic start-up and verification testing of the pilot unit and that this effort will immediately procede pilot plant testing (Figure 1). A schedule, detailing the start-up and operational verification, is presented as Figure 2.

4.1 PERSONNEL TRAINING

The initial task of the start-up operation, personnel training, will have as its primary goal the safe and orderly execution of the first pilot plant start-up and shut-down. To facilitate the achievement of that goal, all operating, laboratory and engineering personnel will be familiarized with all start-up and shut-down procedures, both normal and emergency. The training procedure will consist of operating manual study, safety manual study and on-site inspection of the processing equipment and area to familiarize personnel with types and locations of processing equipment, valves, service facilities, instrumentation and control hardware, effluent monitoring equipment and emergency control points.

As indicated in Figure 2, the above mentioned training program will be carried out over a 1-month time period with the second half of the program overlapping the initial testing of the Coal Feed and Reactor Sections of the process. The familiarization program will, therefore, be organized to

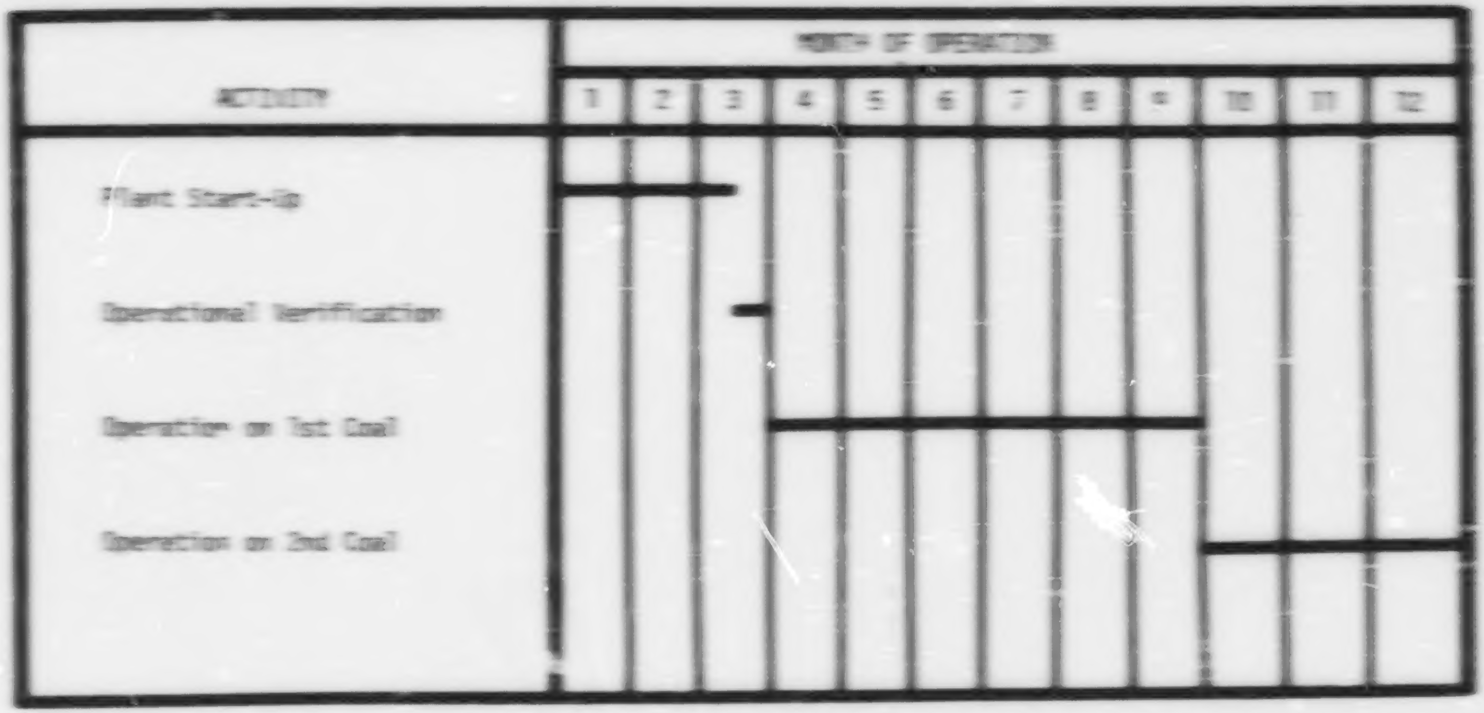

Figure 1. Pilot Plant Operation Schedule

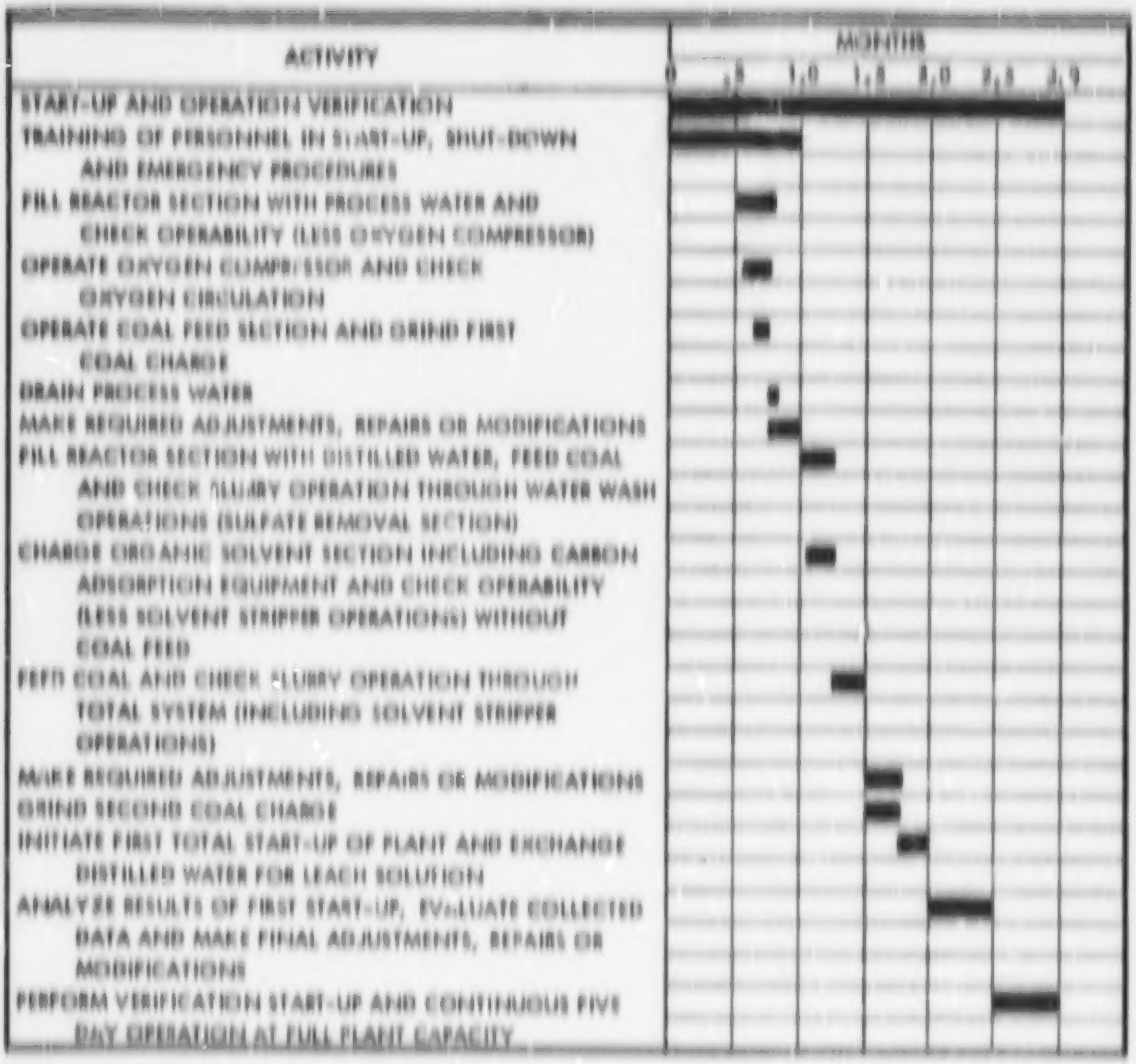

Figure 2. Pilot Plant Start-up and Operational Verification

concentrate all training associated with these two processing sections during the first half of the schedule with the remainder of the processing sections being covered during the overlap period.

4.2 PILOT PLANT START-UP OPERATIONS

Following personnel training, the initial start-ups, short-term operations and shut-downs of the processing sections will be performed. The primary goal of these operations is to check out equipment, personnel, procedures, analytical techniques and overall operability of the integrated system. The initial start-up will require elongated time schedules to allow for greater process control and more in-depth procedure and equipment evaluations than will be required during subsequent start-ups. During plant start-up and subsequent operations, the process will be segregated into the following functional processing sections (equipment numbers are from the flowsheet):

COAL FEED SECTION

Belt Feeder (A-1)
Apron Feeder (A-2)
Pulverizer (A-3)
Bin Discharger (A-4)
Coal Hopper (A-8)
Weigh Belt (A-5)
Storage Tank (T-1)
Dust Filter (S-1)
Dust Blower (B-1)
Rotary Valve (SP-8)

REACTOR SECTION

Primary Reactor (R-1)
Slurry Mixers (M-2a/3)
Knock-Out Drum (V-1)
Oxygen Recycle Compressor (K-1)
Secondary Reactor (R-2)
Cooling Water Drum (T-3)
Feed Pump (P-1)
Secondary Reactor Mixers (M-12,M-13)
Flash Drum (T-4)
Cooling Water Drum (T-5)
Pumps (P-2a/3, P-3)
Feed Mix Tank (T-2)
Feed Tank Mixers (M-1a/c)
Scrubber Blower (B-2)
Scrubber (SP-1)

SULFATE REMOVAL SECTION

Thickener (R-3)
Hydroclone (SP-2)
Make-up Tank (T-6,7)
Thickener Discharge Pump (P-4)
Leach Solution Surge Tanks (T-9,10)
Surge Tank Pumps (P-6,7)
Evaporator Crystallizer (SP-3)
Wash Water Contactors (T-11,13)
Contacter Pumps (P-10,13)
Surge Tank Pump (P-19)
Hydroclone Surge Tanks (T-26,27)
Hydroclone Mixers (M-10,11)
Hydroclone Pump (P-26)

Receivers (V-2,3,4,5,6)
Filtrate Pumps (P-8,11,14)
Make-Up Tank Mixers (M-3,4)
Make-up Transfer Pump (P-5)
Wash Water Pumps (P-9,12)
Filters (S-2,3,4)
Vacuum Pumps (K-2,3,4)
Barometric Condensors (E-1,2,3)
Contactor Mixers (M-6,7)
Waste Disposal Tanks (T-25A,B)
Waste Transfer Pumps (P-25)
Wash Water Surge Tank (T-20)

ORGANIC SOLVENT SECTION

Rotary Feeder (SP-7)
Azeotrope Still (T-14)
Still Mixer (M-8)
Solvent Centrifuges (S-5,6)
Centrate Receiver (T-15)
Solvent Contactor (T-16)
Contactor Mixer (M-9)
Coal Cooler (E-4)*
Solvent Stripper (SP-4)*
Azeotrope Still Pump (P-15)
Centrate Pump (P-16)
Contactor Pump (P-17)
Surge Tank Pumn (P-20)
Waste Disposal Tank (T-24)
Waste Transfer Pump (P-24)

Coal Elevatur (A-6)*
Stripper Condensor (E-5)*
Stripper Decanter (T-18)*
Still Condenser (E-6)
Still Decanter (T-19)
Surge Tank (T-21)
Solvent Surge Tank (T-22,23)
Solvent Still (C-1)
Still Condenser (E-7)
Solvent Cooler (E-9)
Sulfur Filters (S-7,8)
Solvent Surge Tank Pumps (P-21,22)
Solvent Still Pump (P-23)
Carbon Absorption Drum (SP-5)
Screw Conveyor (SP-6)

For purposes of discussion in this report, the above described sections will be referred to as follows:

Section 1 - Coal Feed Section
Section 2 - Reactor Section
Section 3 - Sulfate Removal Section
Section 4 - Organic Solvent Section

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*These items of equipment constitute the solvent stripping operation.

The estimated time required to perform all of the necessary start-ups and shut-downs, including anticipated equipment adjustment, repairs and modifications, is 1-1/2 months. As shown in Figure 2, this effort immediately precedes the operational verification test run. It is anticipated that all initial start-ups will be performed with the secondary reactor (R-2) operating as an atmospheric pressure holding tank, and with all equipment operating at low to moderate capacities on the order of 250 to 500 lbs/hr coal feed equivalent. The following paragraphs present a description of the initial start-up sequence.

During the initial week of start-up operations, the primary and secondary reactors (R-1 and R-2) will be filled to appropriate operating levels with process water (simulating leach solution). The aqueous effluent line from the secondary reactor (stream 13) will be circuited to the leach solution return line (stream 17) during this test operation, thus effecting a closed circulation loop around the knock-out drum (V-1), scrubber mist eliminator (SP-1), slurry feed mix tank (T-2), primary reactor (R-1) and secondary reactor (R-2). Section 2, with the exception of the oxygen circulation equipment, will then be put into operation utilizing ambient temperature process water at atmospheric pressure in the following manner:

(1) Start water flow through all scrubbers and check operability.
(2) Start all mixer motors and check for operability.
(3) Start reactor recirculation pumps and check for operability.
(4) Start slurry feed pump, reactor discharge pump and check operability.

With Section 2 (less oxygen compressor) operating at ambient temperature and pressure conditions, associated equipment such as pressure, temperature, flow and level controllers, valves, recorders and indicators will be checked for integrated operability, as will be all instrumentation circuits, compressor cooling water circuits and seal flush systems, etc. After achieving successful operation of the Reactor Section, with liquid circulating at ambient temperature and pressure, oxygen compressor operations will be initiated and oxygen system pressure will be increased to approximately 50 psig. At this point, such additional items as compressor system integrity and pump seal operation will be checked. Following successful operation of the primary reactor at 50 psig oxygen pressure and ambient temperature, the process water will be steam heated to approximately 250°F where operation will be checked, as will be sampling equipment and techniques. Section 2 will then be shut-down (reverse order of start-up). During the initial start-up of Section 2, no processing data will be collected.

Also scheduled (Figure 7) for operation during the first month of start-up is Section 1. The apron feeder, feeder belt, rotary valve, pulverizer, elevator, weigh belt, dust filter and dust blower will sequentially be started to insure system integrity and operability without coal feed. The nitrogen blanketing system will also be tested to insure operability. During this period all associated equipment will be checked and the weigh belt zero point will be verified. Following the mechanical check of Section 1, coal will be fed onto the apron feeder and transported to the pulverizer where the first grinding operation will take place. The ground coal will then be transported to the coal storage tank (T-1), where it will be held until required for slurry operations. A total of approximately 36 tons of coal will be pulverized to -100 mesh particle size and stored following an initial sampling of the pulverizer product to ensure proper sizing.

During the third week of start-up operations, Section 2 will be drained of process water utilizing the emergency dump lines and waste disposal tanks (T-25A and T-25B). The water will then be pumped from the disposal tanks and returned to the process cooling water pond. This operation will ensure the operability of the emergency dump system under non-slurry conditions. In addition to draining Section 2, any adjustments, repairs or modifications to either Section 1 or 2 (indicated during the first start-up) will be made during the second week of operations. Additionally, the bypass lines between streams 13 and 17 will be removed and the washed coal feed line (stream 33) to the azeotrope still (a solid transport chute) will be diverted into dumpster waste containers. This modification to normal flow will bypass Section 4 and allow simultaneous operation of Sections 1, 2, and 3 of the plant. Also, the wash water line to contactor T-13 (stream 29) will be temporarily connected to the output of the wash water surge tank (stream 53) to allow for complete recirculation of the aqueous streams with no make-up or blow-down.

The second month of start-up operations will begin with the refilling of Section 2 with purchased distilled water (not process water). The reactor-sulfate removal operations will then be started, utilizing ambient temperature distilled water and 50 psig oxygen pressure in the primary reactor. Section 2 will be started as previously discussed. Section 3 equipment will be sequentially started as the fluid (distilled water) flows from one processing unit to another. This sequential start-up will ensure against such problems as "dry dumping" (i.e., pumps operating with no fluids in lines) and "slugging" (i.e., insufficient liquid levels in vessels allowing air to mix with pumping fluid resulting in two-phase flow).

As the distilled water fills the vessels in Section 3, a make-up of fresh distilled water will be fed to Section 2 utilizing the leach solution make-up tanks (T-6 and T-7) and transfer pump (P-5). This make-up will be required until total recycle of the distilled water is achieved, including the recycling of water through all wash streams. During the initial operation of Section 3 with ambient temperature distilled water, all associated equipment such as gauges, controls, valves, sampling equipment, sampling techniques, instrumentation, barometric leg condensers, vacuum pumps, water systems, seal flush systems, etc., will be checked for integrated operability. Also, the secondary processing equipment in Section 3, namely the thickener and hydroclones, will be operated at this point, only to demonstrate mechanical integrity and operability. Following the ambient water temperature operation, the distilled water will be steam heated to appropriate nominal processing temperatures and circulated through Section 3.

After the equipment is checked for operability under elevated temperature conditions, Sections 2 and 3 will be ready to be slurry tested, utilizing Section 1. The weigh belt and rotary valve will be activated and pulverized coal will be fed into the slurry feed mix tank (T-2). A 20% coal-distilled water slurry will be mixed and fed to the primary reactor. During this mode of operation, special care will be taken to follow the advance of the slurry through the process (over an approximately 2-1/2 day period) with careful attention being paid to all rotary equipment seeing slurry service for the first time. During this start-up, an indication of the time required for the slurry to reach steady concentration levels in each processing unit will be obtained. As slurry operation is attained, all pumps will be checked for turn-down limits and bypass loop requirements will be determined. Filters and their internal wash-water spray systems will be evaluated for operability and all solids transfer operations will be checked. The wet coal cake exiting the third filter (S-4) will be collected in dumpsters (utilizing a solids diverter valve in the coal cake transport chute), sampled, and trucked to disposal sites. After steady state operation has been reached in Sections 1, 2, and 3, operating parameters will be recorded and samples will be withdrawn, analyzed, recorded and reported as a water blank, noting such things as sulfate build-up in the water (water soluble sulfate leaching from the coal).

Section 4 will undergo its first start-up simultaneously with Section 3 during the second week (Figure 2). The primary objective of this initial start-up is to check liquid toluene circulation operations without introducing pulverized coal. During its initial start-up, this section will be separated and operated in a closed loop fashion. This mode of operation will be accomplished with the slurry feed port of the azeotrope still (T-14) sealed. Also, since no coal will be fed into the section, the solvent wet coal cake transport chute between the last centrifuge (S-6) and solvent stripper (SP-4) will be valved shut. The solvent stripper operations (SP-4, E-4, A-6, E-5, T-18) will not be started up until the next run which will include process slurry operations.

Prior to operation, the carbon absorption drum (SP-5) will be charged and the entire section will be purged with an inert gas (N_2). Extreme care will be taken to ensure against the occurrence of flammable mixtures (toluene-air) in this processing section. The azeotrope surge tank (T-21) and rich solvent surge tank (T-22) will then be charged with toluene. The transfer pump will then be started and the remaining equipment will be filled with solvent to the appropriate level. Make-up toluene will be continuously fed into the lean solvent surge tank (T-23) until continuous recycle of ambient temperature toluene is achieved. The section will be checked for operability and integrity under ambient operation conditions and then steam header and cooling water circuits will be put into operation, thus bringing the Organic Solvent Section to nominal processing temperature operation. While circulating process temperature toluene, all associated equipment such as level, temperature and pressure control equipment, instrumentation, lubrication systems, pump and mixer seals, sampling devices and techniques, etc., will be checked for operability. At this point, the process sections in operation will be shut down and the coal cake transfer chute between the final filter unit and the azeotrope still (stream 33) will be reconnected as will be the transfer chute between the last centrifuge and the solvent stripper (stream 44).

The total processing unit, including the solvent stripper operations, will then be readied for start-up with coal slurry feed throughout the system (utilizing water as the leaching solution) during the fourth week of operations. Prior to this start-up, a thorough check-out of the nitrogen recirculating and blanketing system associated with the stripper unit will be conducted to ensure that all safety devices are operable. Following that precautionary measure, the total system will be started up sequentially. The slurry advance through Section 4 will be closely monitored to ensure operability of all rotating equipment seeing slurry service for the first time. Also, start-up of the stripper unit will be carefully watched to ensure maximum safety of that potentially dangerous first-time operation. After steady-state operation has been attained, feed, water, solvent, slurry and product coal samples will be collected, analyzed and reported as blank run results (utilizing distilled water as substitute leach solution in Section 2). During this run, the product coal will be collected in dumpsters and trucked to disposal. The pilot unit will then be shut down.

During the fifth week of operation, the pilot plant will undergo any repairs, adjustments or modifications to Section 2 and/or Section 4 which were indicated during test runs. Also, the previously modified wash water supply lines allowing total recirculation of wash water (detailed in the Sulfate Removal Section start-up discussion) will be reconnected for normal process operations. This mechanical work may require some equipment to be drained. If this is the case, the equipment will be drained into the appropriate waste disposal tanks for storage (T-24, T-25A, T-25B), depending upon the nature of the fluid (i.e., organic or aqueous). The adjustments, repairs or modifications will then be made and the fluids pumped back to their point of origin utilizing transfer pumps (P-24, P-25). Also, during the fifth week of operation, a second charge of coal (approximately 30 tons) will be pulverized and stored in the coal storage tank (T-1). The pilot plant will then be readied for the next start-up.

During the sixth week of start-up operations, the pilot plant will undergo an additional start-up and short-term (2 to 3 days) operation for the purpose of exchanging the distilled water in the Reactor Section with leach solution. The exchange will be accomplished by feeding ferric sulfate crystals directly into the feed-mix tank (T-2) through the pulverized coal feed mechanism. The operation will be carried out so as to leave the reactor system filled with dilute leach solution (approximately 2%) and ready for the verification test and subsequent pilot plant operation.

During the start-up, all equipment seeing leach solution for the first time will be checked for operability in that service. Special attention will be paid to mechanical seals and seal water flush systems. As the distilled water is exchanged for leach solution, samples will be collected and data obtained on plant operation throughout the unit. This information will serve as baseline data and indicate transient operational effects and time requirements. The plant will be operated long enough to ensure that steady-state operation is effected and then shut down. During this start-up operation,the product coal and byproduct sulfate and sulfur will be collected and temporarily stored at the test site. The products will eventually be trucked to disposal.

During the seventh and eighth weeks of operation, the initial start-up procedures and all operational data gathered during previous runs will be thoroughly analyzed as will be sample analysis results. The test data will be analyzed with respect to the units capability of sustaining continuous operation and attaining desired processing objectives. Additionally, the detailed test plan for initial pilot plant operation will be reviewed and modifications submitted for approval by EPA, if the start-up test data and/or operating experiences indicate changes in test sequence or schedule are desirable. Also, during this time period, any further adjustments, repairs, or modifications to the processing unit will be made.

The plant will then be readied for a 5-day, full capacity (1000 lb/hr coal feed) verification start-up and continuous operation test. Additional coal will also be pulverized and the coal feed storage tank (T-1) filled to capacity.

4.3 PILOT PLANT OPERATIONAL VERIFICATION

During the final 2 weeks of the start-up and verification operations (Figure 2), the pilot unit will be started up and continuously operated for a 5-day test period at full design capacity. The probable processing parameters to be utilized during this operation are:

Coal feed rate = 1000 lb/hr
Coal particle size = -100 mesh
Slurry concentration = 20% coal
Leach solution concentration = 2% iron
Reaction-regeneration temperature = 225°F
Oxygen pressure = 50 psig
Excess oxygen circulation = 4 x consumption

The time period required for the system to reach steady-state operation (after start-up) will be about 2-1/2 days. This corresponds to the residence time of the coal in the system from coal feed storage tank (T-1) through the solvent stripping unit (SP-4). The unit will then be operated for approximately 2-1/2 days at full capacity. During the start-up operation, the system throughput will be increased from the 250 to 500 lb/hr of coal feed equivalent previously utilized to 1000 lb/hr. This will require that additional leach solution, of appropriate concentration, and toluene be fed into the unit during simultaneous increase of coal feed. During the course of the operation fresh coal will be pulverized so as to maintain continuous coal feed. Data on transient process operation will be obtained during this operation. While in operation, complete sampling procedures will be performed at all sampling points. All monitored operational data will be recorded. The operational data collected and sample analysis results obtained will be analyzed as soon as possible so that on-line operational adjustment can be made during the course of the test. All data will be analyzed, recorded and reported following the test run and the pilot plant will be approved for pilot operations.

At the conclusion of the test run, the process unit will be shut down with all solid process effluents being collected and temporarily stored at the site and liquid waste effluents being trucked to disposal. Results obtained during the verification test will be reviewed and changes in equipment and procedure will be made where deemed necessary.

5. PILOT PLANT OPERATION

Following start-up and operational verification, the pilot plant will be put into operation to characterize process operability on two coals. The general philosophy of plant operation will be to operate initially on one coal to completion of a test matrix aimed at characterizing both the unit operations within the plant and the applicability of the coal being tested. This operation is expected to require approximately 6 months of testing with an additional month of data evaluation. The second phase of the test program will involve the second coal and require an estimated 3 months for completion. During the second phase of the test program, emphasis will be placed on determining the coal's applicability to the process. Much less effort (than in the first part of the test program) will be required to characterize unit operations within the plant since a large number of those operations are expected to be substantially independent of coal type. Also, during the entire test program, the operational and process chemistry testing to be performed will be segregated into three categories: (1) primary test variables, (2) secondary test variables, and (3) supportive process monitoring. The primary test variables are those which are independent of process operation and will be varied for study throughout a given test run. The secondary test variables are those which are dependent upon the primary variables and change when changes in the primary variables occur. For instance, the slurry concentration (coal/leach solution) being evaluated in the primary reactor (R-1) is a primary variable while this variable's resultant effect upon operation of the coal feed-mix tank (T-2), secondary reactor (R-2) and the first filter (S-2) is a secondary test variable for which data will be collected and evaluated. Supportive process monitoring is that monitoring required to ensure desired, safe, and environmentally acceptable process operation. The primary objective of the pilot plant test program is to prove out the technology for chemical desulfurization of coal in an integrated, continuous, process system. That objective will be accomplished by (1) evaluate parametrically each processing section and/or major piece of equipment with respect to its operability on the subject coals, (2) thoroughly characterize all feed

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materials, coal products, sulfur and sulfate by-products and all other effluent streams, and (3) generate sufficient quantities of products and by-products for future large-scale testing evaluation.

The data obtained during the performance of the pilot plant studies will be utilized as a foundation for:

(1) Recommendation of techniques for efficient operation and design optimization of the process while achieving maximum sulfur removal.

(2) Recommendation of techniques for minimizing capital and operational costs while maintaining optimum sulfur removal.

(3) Recommendation of methods for ensuring environmental integrity and safe operability of the process.

(4) Generation of up-dated pilot plant flow sheets and equipment and material specifications with appropriate continuous operation mass and energy balances.

(5) Generation of data to permit performance of sound process design and economic studies.

(6) Development of the methodologies for effective process system measurement and monitoring.

The following paragraphs describe in detail how the above mentioned objectives will be met with respect to each major section of the pilot plant (pilot plant sections as defined in Section 4.3 of this document) and present an anticipated test matrix and schedule of operation for utilization during evaluation of the two coals.

5.1 EQUIPMENT TESTING AND ANALYSIS REQUIREMENTS

Presented in this section is a detailed description of the operating parameters to be evaluated for the equipment in each of the four major processing sections of the pilot plant. Additionally, chemical analysis requirements associated with operational or process chemistry evaluations are discussed.

5.1.1 Coal Feed Section Testing

The primary functions of this section are to deliver coarse coal, from the coal storage pile to the pulverizing equipment (see process flow diagrams in Appendix A) and transport the pulverized coal to the pulverized coal storage tank (T-1). Since it is anticipated that the mechanical operability of all equipment in this section will have previously been demonstrated during start-up and verification operations, the only testing to be performed in this section will be that of obtaining pulverizer input and output and performing sieve analysis to ensure proper particle size feed to the reactor section and periodically monitoring the pulverizer system purge gas effluent for particulate.

5.1.2 Reactor Section Testing

In this section of the process the pulverized coal is mixed and wetted with leach solution while maintaining minimum foam generation. Also, the pyrite contained within the coal is converted to elemental sulfur and soluble iron sulfate and regeneration of the spent leach solution takes place.

Anticipating that mechanical operability of all equipment will have been previously demonstrated during pilot plant start-up and check-out, the only equipment in this section requiring thorough operational characterization is the slurry feed-mix tank (T-2), the scrubber-mist eliminator (SP-1), the primary reactor (R-1), and the secondary reactor (R-2). The secondary reactor (R-2) will be characterized while operating both as a holding tank (also simulating a large thickener), a true secondary reactor with no regeneration taking place, and as scaled-up (approximately 10 times larger than R-1) primary reactor with regeneration.

Operational variables to be evaluated during reactor operation include (1) coal characteristics, (2) coal particle size, (3) slurry residence time (1-10 hours, controlled through slurry feed pump (P-1) rate and number of reactor sections utilized), (4) reaction temperature (220°F-275°F), controlled by varying inlet slurry temperature and steam injection, (5) oxygen partial pressure and circulation rates, varied through changes in compressor recycle, oxygen bottle pressure and blow-down pressure setting, (6) mode of three phase (oxygen-coal-leach solution) mixing, which will be evaluated utilizing oxygen injection in slurry pump-around loops, static mixing and oxygen injected through gas dispersion agitators, (7) slurry concentration (coal/leach solution ratio), and (8) salt concentration in solution. The parameters to be evaluated during slurry feed-mix tank (T-2) operation are (1) coal characteristics, (2) coal particle size, (3) slurry residence time (15 min to 1 hr) which will be varied through the use of liquid level control and feed rate, (4) slurry temperature (180°F-215°F) which will be varied by steam injection, and (5) mixer horsepower requirements.

Sampling requirements during reactor (both R-1 and R-2) operation will include slurry samples drawn from each reactor section of both reactors, with the coal being separated and analyzed for pyrite and elemental sulfur to determine degree of pyrite conversion to sulfur and sulfate, and leach solution being analyzed for ferric and ferrous ion to determine degree of regeneration. The reactor (R-1 and R-2) liquid effluent (slurry) will also be analyzed for trace materials to evaluate the problem of impurities build-up in the leach solution due to recycling. The gaseous effluent from the primary reactor (R-1) will be sampled prior and subsequent to the knock-out drum (V-1) and analyzed for particulate and the presence of foam. Sample requirements for slurry feed-mix tank (T-2) characterization include (1) pulverized coal feed sampling to determine particle size distribution, (2) coal slurry sampling from the feed-mix tank effluent (from individual stages) which will be analyzed for pyrite conversion and examined to determine degree of wetting and general slurry composition, and (3) gas sampling from gas effluent lines running from the feed-mix tank to the scrubber mist eliminator (SP-1) which will be analyzed for carbon dioxide content (carbon dioxide must be evolved prior to entrance into the reactor section in order to minimize oxygen bleed requirements) and foam content.

In addition to the above, the effluent from the scrubber-mist eliminator (SP-1) will be monitored for the presence of coal and acid mists and the gaseous effluent from the blower (B-2) and aqueous effluent from the cooling water drum (T-3) will be sampled and analyzed for particulate and sulfate presence. The oxygen blow-down will be sampled and analyzed for impurities build-up with oxygen consumption then being determined by mass balancing around the reactor (R-1). Also, the cooling water drum (T-5) effluent will be sampled and analyzed for particulate and acid content.

6.1.3 Sulfate Removal System Testing

The equipment in this section requiring thorough operational characterization are the thickener (R-3), hydroclone (SP-2), evaporator-crystallizer (SP-3), filters (S-2,3,4) and wash-water contactors (T-11,13). The remainder of the equipment will require minimal characterization since mechanical operability will have previously been demonstrated during start-up.

The thickener (R-3) will be operated either off-line or in parallel to the secondary reactor (R-2). Its ability to thicken the coal slurry, and hence lower subsequent filtering duty, will be evaluated as a function of (1) coal characteristics and particle size, (2) slurry residence time, (3) inlet temperature and (4) slurry concentration (coal/leach solution ratio). This evaluation will require sampling of thickener effluents (overflow and concentrate) and determination of coal-to-leach solution ratios. The hydroclone (SP-2) will be operated in series with the secondary reactor (R-2). Its ability to thicken the coal slurry will be evaluated as a function of coal characteristics, slurry flow rate (i.e., residence time in the hydroclone), slurry composition (coal/leach solution ratio), slurry temperature and coal particle size. This characterization will be accomplished by sampling hydroclone effluents (overflow and concentrate) and determining coal-to-leach solution ratios.

The evaporator-crystallizer (SP-3) will be operated off-line and be evaluated with respect to output product (crystalline iron sulfate salts) characteristics as a function of operation parameters. Variables which will be studied include (1) input leach solution composition, (2) feed rate, (3) operating pressure (sub-atmospheric to one atmosphere), and (4) concentrate circulation rates. Evaluation of the unit will require that concentrate samples be taken and that the crystalline product be analyzed and chemically characterized. Quantities of this by-product will be retained for future study. Samples of the barometric condenser effluent will also be analyzed for particulate and sulfate presence for determination of any possible environmental impact.

The three filter stages (S-2,3,4) in this processing section will each be characterized separately since they represent three different types of filtration equipment (as discussed earlier in Section 3.0). Each filter will be evaluated while operating in each of the three positions of the filtration sequence. Filtration and wash efficiencies of each unit will be determined as a function of (1) input slurry concentration and temperature, (2) coal characteristics and particle size, (3) drum speed (i.e., cake thickness), (4) wash-water feed rate, (5) suction pressure, and (6) filter cloth type.

In general, all filter influents and effluents will require sampling and analysis for complete characterization. The slurry feed, filtrate, spent solution and filter cake will be analyzed for iron sulfate to evaluate wash and filtration efficiencies and determine the fate of the soluble salts. Additionally, the moisture content of the cake will be determined. The wash water will also be analyzed for sulfate if it is not fresh, unrecycled (uncontaminated) water. Also, the effluents from the vacuum pumps (K-2,3,4) and barometric condensers (E-1,2,3) will be sampled and analyzed for particulate and sulfate presence for evaluation of possible adverse environmental impact.

The wash-water contactors (T-11,13) will be evaluated with respect to their ability to induce dilution of the soluble sulfate within the coal matrix. Operational variables to be evaluated include (1) input cake and diluent composition, (2) temperature, (3) coal characteristics and particle size, (4) residence time, and (5) mixer horsepower input. Samples will be obtained from input and output streams with the coal being separated from the slurry and analyzed for sulfate presence to determine the degree of sulfate concentration reduction in the coal matrix.

5.1.4 Organic Solvent Section Testing

The primary functions of this section of the process are to effect a separation and recovery of the elemental sulfur from the coal matrix, recover the organic solvent utilized in the extraction, and dry the product coal. The equipment in this section requiring complete operational characterization are (1) the azeotrope still (T-14), (2) the solvent centrifuges (S-5,6), (3) the solvent contactor (T-16), (4) coal cooler (E-4), (5) the solvent stripper (SP-4), (6) the solvent still (C-1), (7) the solvent cooler (E-9), (8) the sulfur filters (S-7,8), and (9) the carbon absorption drum (SP-5). The remainder of the equipment in this section are auxiliaries to the major equipment cited and require only mechanical operability check-out during the start-up phase of this program.

The azeotrope still (T-14) will be evaluated with respect to its ability to effect essentially total displacement of the absorbed water on the feed coal cake with toluene. Variables to be evaluated include (1) coal characteristics, (2) coal particle size, (3) residence time, (4) cake composition, (5) cake/toluene ratio, (6) mixer horsepower requirements, and (7) heat transfer requirements. Samples of the overhead will be collected and analyzed for toluene and water content and organic impurity build-up due to recycling. The bottom product, a coal toluene slurry, will be sampled with the coal being separated and analyzed for moisture content, elemental sulfur content and sulfates. This data will indicate the efficiency of the organic wetting.

The operation of the solvent centrifuges (S-5,6) will be evaluated from the standpoint of washing and filtering efficiency. Variables to be evaluated include (1) input slurry composition and temperature, (2) coal characteristics and particle size, (3) centrifuge rotational speed, and (4) wash solvent feed rate. The feed slurry and wash solvent will be analized for composition, elemental sulfur and sulfate content. The centrate and product coal cake will be analyzed for coal to toluene ratio, elemental sulfur and sulfate content. This data will indicate the wash and filtration efficiences of centrifuges in this application.

The solvent contactor (T-16) will be evaluated with respect to its ability to promote dilution of the organic soluble elemental sulfur within the coal matrix. Operational variables to be tested include (1) input cake and diluent composition, (2) temperature, (3) coal characteristics and particle size, (4) residence time, and (5) mixer horsepower requirement. Samples will be obtained from input and output streams with the coal being separated from the slurry and analyzed for elemental sulfur content to determine the degree of dilution obtained during this operation.

The solvent stripper (SP-4) is essentially a tray drier with solvent recovery capability. The drying medium is primarily nitrogen but may also be steam. The operability of this processing unit will be determined as a function of (1) input cake composition (i.e., solvent content), (2) coal characteristics and particle size, (3) feed rate and temperature, (4) solids residence time (i.e., tray rotational speed), and (5) stripping media quantity and temperature. Cake input and product samples will be analyzed to determine solvent and moisture content in order to evaluate the drying efficiency of the unit. Inlet and outlet temperature will also be monitored.

The coal cooler (E-4) is essentially a water-cooled, hollow-screw type cooler. Its ability to cool the product coal to safe-handling temperatures will be determined. Variables to be checked include (1) cooling water rates, (2) screw rotation speed (i.e., residence time), (3) inlet coal temperature, and (4) coal characteristics and particle size. Inlet and outlet coal temperatures will be monitored and samples of the product coal will be analyzed for elemental sulfur and sulfate to determine overall sulfur removal efficiency of the process. Also, the physical characteristics and toluene retention of the coal will be determined. The product coal will be stored for future use.

The solvent still (C-1) is designed to concentrate the sulfur-rich toluene solvent from about 2.0 wt% sulfur to about 10 wt% sulfur. The operation of this unit will be characterized by varying feed compositions (i.e., sulfur content), feed temperature, and heat duty. Samples of feed and product streams will be analyzed for sulfur as well as other organo-sulfur compounds, such as H_2S, which might form during operation. Also, any heavy bottoms product, which might form during continuous operation with recycling, will be sampled and characterized.

The solvent cooler (E-9) is a scraped surface heat exchanger. This unit's operability will be characterized by determining its ability to promote sulfur crystallization as a function of (1) feed solvent sulfur content and feed rate, (2) cooling water temperature, (3) feed rate, (4) inlet solvent temperature and (5) scraper rotation speed. Samples of the inlet solvent stream will be analyzed for dissolved sulfur content, while effluent solvent will be analyzed for dissolved sulfur as well as crystallized sulfur content. The crystallized sulfur will then be chemically characterized. This data will indicate the degree and form of crystallization which takes place during this operation.

The sulfur filters (S-7,8) are leaf-type filters. The primary objective of their operation is to remove all crystallized sulfur by-product from the recirculating solvent. The operation of the filters will be evaluated as a function of (1) feed solvent composition, (2) sulfur crystal content, (3) feed rate and temperature, and (4) filter cloth type. Samples of the sulfur-crystal-containing feed solvent and the filtrate will be analyzed for sulfur content to determine the filtration efficiency of this unit.

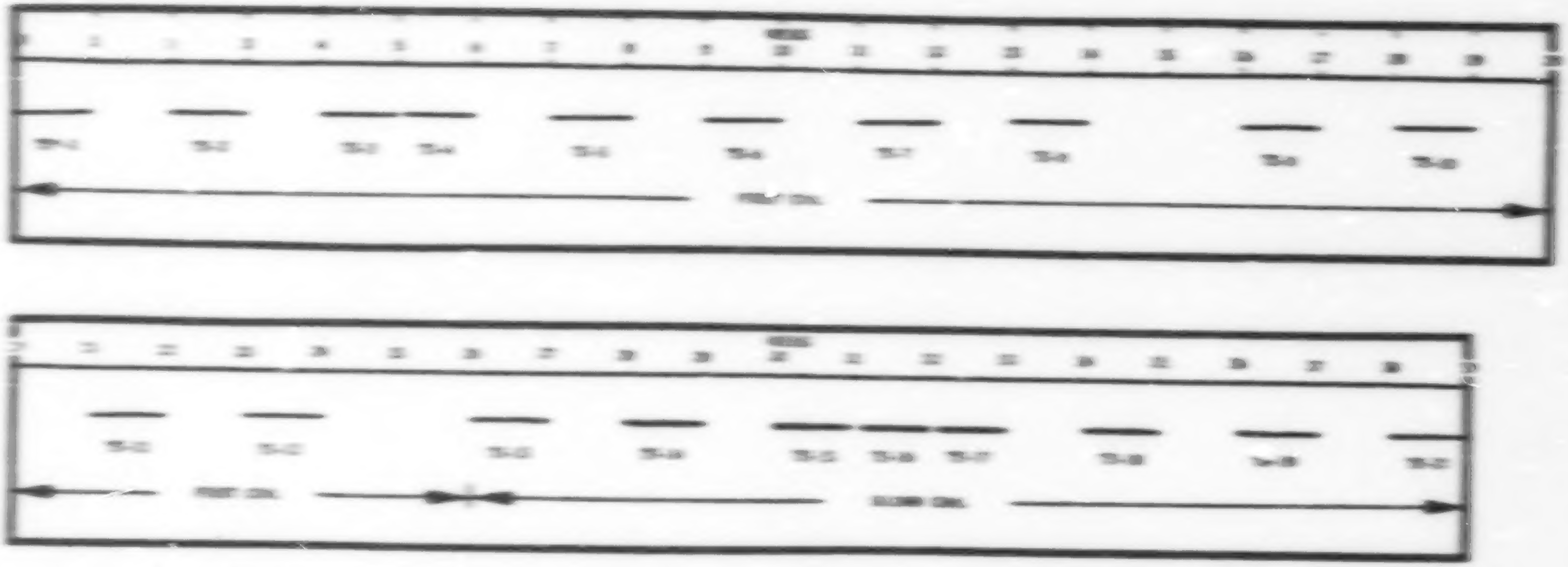
The solid sulfur by-product will be sampled and analyzed for toluene content, other impurities content, and sulfur form. Quantities of the dry product will be stored for future use in application studies.

The carbon adsorption drum's (SP-5) primary function is to handle waste organic vapors and ensure environmental integrity of the plant surroundings. The input and effluent from the carbon drum will be monitored for hydrocarbon presence so that any breakthrough can be detected and a fresh charge of carbon be put into operation.

6.2 Plant Operational Schedule and Test Matrix

The pilot plant test program is scheduled over a 9-month period. The first 6 months of the test program will be devoted to the evaluation of one type of coal while the remaining 3 months will be utilized to evaluate a second type of coal. The testing period will be further broken into individual test runs or test sequences (TS). It is anticipated that 20 test sequences will be run during the entire test program with the initial 12 being concerned with the evaluation of the first coal type and the final eight concerned with the second coal type. The anticipated schedule of operation is presented in Figure 3. The general philosophy behind the schedule is as follows:

- All test sequences except TS-4 and TS-16 involve complete process operation less the off-line evaporator-crystallizer (SP-3).
- TS-4 and TS-16 involve only the evaporator-crystallizer (SP-3) and its support equipment.
- Each TS will require approximately 1 week of operation. Following each sequence, there will be 1 week to evaluate data, make required repairs, adjustments and modifications for the next TS.
- Test sequences involving only the evaporator-crystallizer (SP-3) and its support equipment will be run on "off weeks".



*Test Sequence Number.
Individual tests are detailed
in Table 3.

Figure 3. Anticipated Nine-Month (39 Week) Test Schedule

- An additional "off week" has been added to the schedule periodically (6th, 15th, 20th and 25th weeks) to allow for schedule slippage due to (1) unforeseen occurrences, such as equipment failure, or longer than expected time requirements to complete specific operation studies, (2) the running of additional sequences if operating experience indicates the need, or (3) the rerunning of previous sequences or specific unit operations if the operating data collected were inconclusive.
- The additional "off weeks" are only scheduled during the initial 6 months of operation (the first coal type) since it is anticipated that most of the "surprises" will occur during that time. Previous operating experience should help in anticipating the prevention of such occurrences during the last 3 months of operation.

Based on previous pilot plant operating experience, the above outlined philosophy should result in a realistic rather than optimistic test schedule.

During the course of operation, virtually every piece of equipment in the processing scheme, including several alternates, will be thoroughly characterized with respect to Meyers Process applicability. Table 3 presents a summary of the equipment to be tested and the operational variables to be evaluated. As mentioned previously (Section 6), the variables are categorized as primary (independent) and secondary (dependent). A third type of operation indicated on Table 3 is that of supportive process monitoring to ensure acceptable plant operation. It is intended that throughout any test sequence, all variables (both primary and secondary) will be monitored and that specific primary (independent) variables will be varied and the resultant changes in overall operation (including all dependent variables) evaluated. Also, supportive process monitoring activities will be carried out during every test sequence.

A test program has been prepared and is presented in Table 4 in the form of 20 test sequences. The test sequences are, for the most part (all but TS-4 and TS-16), built around the testing of the reactor section of the process (specifically the primary reactor). This was done since the

TABLE 1. TEST VARIABLES TO BE EVALUATED DURING OPERATION

[illegible]

TABLE 6. TEST SEQUENCE DETAIL

Test Sequence 1 - First Coal

Reactor, R-1

Coal particle size : -100 mesh
Percent iron in leach solution : 25
Slurry concentration : 20% coal
Reaction temperature : 230°F
Feed rate : 250 lb/hr coal equivalent
Oxygenation mode : pump around

Primary Variable

- Oxygen pressure : 50 psig 80 psig, 100 psig

Slurry Mix Tank : T-3

Primary Variables

- Slurry temperature effects
- Mixer power effects
- Slurry residence time effects

Filters, S-2, S-3, S-4

Primary Variables

- Drum speed effects
- Suction pressure effects
- Wash water feed rate effects
- Filter type effects

Test Sequence 2 - First Coal

Reactor, R-1

Coal particle size : -100 mesh
Percent iron in leach solution : 25
Slurry concentration : 20% coal
Feed rate : 250 lb/hr coal equivalent
Oxygenation mode : pump around
Oxygen pressure : 100 psig

Primary Variable

- Reaction temperature : 250°F, 265°F

Cake Water Contactors, T-11, T-13

Primary Variables

- Feed cake/diluent ratio effect
- Mixer horsepower effects

Solvent Contactor, T-16

Primary Variables

- Cake/toluene ratio effects
- Mixer horsepower effects
- Residence time effects

Sulfur Filter, S-7, S-8

Primary Variable

- Feed solvent composition effects

Filters, S-2, S-3, S-4

Primary Variable

- Filter type effect

Test Sequence 3 - First Coal

Reactor, R-1

Coal particle size : -100 mesh
Percent iron in leach solution : 25
Slurry concentration : 20% coal
Reaction temperature : 265°F
Oxygenation mode : pump around
Oxygen pressure : 100 psig

Primary Variable

- Feed rate : 500 lb/hr, 1000 lb/hr coal equivalent

Acetone Still, T-18

Primary Variables

- Residence time effects
- Mixer horsepower effects
- Cake/solvent ratio effects
- Heat transfer rate effects

Solvent Centrifuges, S-5, S-6

Primary Variables

- Rotational speed effects
- Wash liquor feed rate effects

Filters, S-2, S-3, S-4

Primary Variable

- Filter type effects

Solvent Still ST-1

Primary Variable

- Heat Transfer rate effects

Test Sequence 4 - First Coal

Evaporator-Crystallizer, EF-1

Feed : rich leach solution obtained during TS-1
and stored in storage tank T-25A or B.

Primary Variables

- Feed rate : 250 lb/hr, 1000 lb/hr coal equivalent
- Concentrate circulation rate
- Operating pressure : atmospheric, vacuum

TABLE 4 (Continued). TEST SEQUENCE DETAIL

Test Sequence 5 - First Coal

Reactor, R-1

Coal particle size : -100 mesh
Slurry concentration : 20% coal
Reaction temperature : 250°F
Oxygen pressure : 100 psig
Oxygenation mode : pump around
Feed rate : 500 lb/hr coal equivalent

Primary Variable

- Percent iron in leach solution : 2.5%, 5%

Filters, F-2, F-3, F-4

Primary Variables

- Wash water feed rate effects
- Washing with dilute leach solution effects

Coal Cooler, E-4

Primary Variable

- Coal residence time effects

Test Sequence 6 - First Coal

Reactor, R-1

Coal particle size : -100 mesh
Percent iron in leach solution : 5%
Reaction temperature : 250°F
Oxygen pressure : 100 psig
Oxygenation mode : pump around

Primary Variables

- Slurry rate : 40% coal
- Feed rate : 500 lb/hr, 1000 lb/hr coal equivalent

Filters, F-2, F-3, F-4

Primary Variables

- Drum speed effects
- Suction pressure effects
- Wash water flow rate effects

Solvent Cooler, E-5

Primary Variable

- Scraper rotation rate effects

Slurry Mix Tank, T-1

Primary Variables

- Mixer horsepower effects
- Slurry temperature effects

Test Sequence 7 - First Coal

Reactor, R-1

Coal particle size : -100 mesh
Percent iron in leach solution : 5%
Reaction temperature : 250°F
Oxygen pressure : 100 psig
Slurry concentration : 20% coal
Feed rate : 500 lb/hr coal equivalent

Primary Variable

- Oxygenation mode : pump around, static mixer, direct induction, static head

Test Sequence 8 - First Coal

Reactor, R-2 (operating as scaled-up primary reactor)

Coal particle size : -100 mesh
Percent iron in leach solution : 5%
Slurry concentration : 20% coal
Reaction temperature : 250°F
Oxygen injection mode : pump around
Feed rate : 1000 lb/hr coal equivalent

Primary Variable

- Oxygen pressure : 50 psig, 100 psig

Thickener, ST-1

Primary Variables

- Slurry flow rate effects
- Slurry temperature effects

TABLE 4 (Continued). TEST SEQUENCE DETAIL

[illegible]

TABLE 4 (Continued). TEST SEQUENCE DETAIL

Test Sequence [illegible] - Second Coal

Reactor, [illegible]

Coal particle size - 100 mesh
[illegible] - 35
[illegible]
[illegible] coal equivalent
[illegible]
[illegible] temperature - [illegible]°F

Primary Variable

- Oxygen pressure - [illegible] psig, [illegible] psig, 100 psig

Slurry Mix Tank, [illegible]

Primary Variables

- Slurry temperature effects
- [illegible]
- Slurry residence time effects

Thickener, [illegible]

Primary Variables

- Residence time effects
- Slurry temperature effects

Hydroclone, [illegible]

Primary Variables

- Slurry flow rate effects
- Slurry temperature effects

Solvent Stripper, [illegible]

Primary Variables

- [illegible] temperature effects
- [illegible] rate effects
- Residence time effects

Coal Chute, [illegible]

Primary Variables

- Coal residence time effect

Test Sequence [illegible] - Second Coal

Reactor, [illegible]

Coal particle size - 100 mesh
[illegible] - 35
[illegible]
[illegible] equivalent
[illegible]
Oxygen pressure - 100 psig

Primary Variable

- Temperature - [illegible]°F, [illegible]°F

Filters, [illegible]

Primary Variables

- [illegible] effects
- [illegible]
- [illegible] pressure effects

Solid Bowl Centrifuges, [illegible]

Primary Variables

- [illegible] speed effects
- [illegible] time effects

Test Sequence [illegible] - Second Coal

Reactor, [illegible]

Coal particle size - 100 mesh
[illegible] - 35
[illegible]
[illegible]
[illegible]
Oxygen pressure - 100 psig

Primary Variable

- Feed rate - 500 lb/hr, 1000 lb/hr coal equivalent

Acceptance Tests, [illegible]

Primary Variable

- [illegible] effects
- [illegible]
- [illegible]
- Feed [illegible] rate effects

Test Sequence [illegible] - Second Coal

[illegible], [illegible]

Feed - [illegible] solution obtained during
[illegible] in storage tank [illegible]

Primary Variables

- Feed rate - 250 lb/hr, 500 lb/hr, 1000 lb/hr
[illegible]
- [illegible]
- Operating pressure - atmospheric, varied

TABLE 4 (Continued). TEST SEQUENCE DETAIL

Test Sequence 17 - Second Coal

Reactor, R-1

Coal particle size : -[illegible] mesh
Percent iron in leach solution : [illegible]
Slurry concentration : [illegible] coal
Oxygen pressure : 100 psig
Oxygenation mode : pump around
Feed rate : 500 lb/hr coal equivalent

Primary Variable

● Reaction temperature : [illegible]°F, [illegible]°F, [illegible]°F

Filters, [illegible]

Primary Variables

● Drum speed effects
● Vacuum pressure effects
● Wash water feed rate effects

Solvent Centrifuges, S-5, S-6

Primary Variables

● Rotational speed effects
● Wash solvent feed rate effects

Test Sequence 18 - Second Coal

Reactor, R-1

Coal particle size : -[illegible] mesh
Percent iron in leach solution : [illegible]
Slurry concentration : [illegible]
Oxygenation mode : pump around
Feed rate : 500 lb/hr coal equivalent
Oxygen pressure : 100 psig

Primary Variable

● Reaction temperature : [illegible]°F, [illegible]°F, [illegible]°F

Solvent Centrifuges, S-5, S-6

Primary Variables

● Rotational speed effects
● Wash solvent feed rate effects

Solvent Stripper, SF-4

Primary Variables

● Operational temperature effects
● Residence time effects
● Stripping media circulation rate effects

Test Sequence 19 - Second Coal

Reactor, R-1

Coal particle size : -[illegible] mesh
Percent iron in leach solution : [illegible]
Slurry concentration : [illegible]
Oxygenation mode : pump around
Feed rate : 500 lb/hr coal equivalent
Reaction temperature : [illegible]°F

Primary Variable

● Oxygen pressure : [illegible] psig, [illegible] psig

Thickener, [illegible]

Primary Variables

● Residence time effects
● Slurry temperature effects

Hydroclones, SF-2

Primary Variables

● Slurry flow rate effects
● Slurry temperature effects

Test Sequence 20 - Second Coal

Reactor, R-1

Coal particle size : -[illegible] mesh
Percent iron in leach solution : [illegible]
Slurry concentration : [illegible]
Oxygenation mode : pump around
Feed rate : 500 lb/hr coal equivalent
Reaction temperature : [illegible]°F

Primary Variable

● Oxygen pressure : [illegible] psig, [illegible] psig

Thickener, [illegible]

Primary Variables

● Residence time effects
● Slurry temperature effects

Hydroclones, SF-2

Primary Variables

● Slurry flow rate effects
● Slurry temperature effects

Solvent Stripper, SF-4

Primary Variables

● Operational temperature effects
● Stripping media circulation rate effects
● Residence time effects
● Steam vs nitrogen operation effects

Coal Cooler, [illegible]

Primary Variable

● Coal residence time effects

characterization of process chemistry and reactor design is considered of primary importance to this project and also because of the long residence times required in that processing section. The test sequences are set up to evaluate process chemistry and characterize equipment operation. The proposed test program is likely to undergo modifications as plant operating experience indicates the need for additional or repeated equipment or process chemistry testing. Also, schedule rearrangement may become necessary if the need to test some specific operation (scheduled for later testing) becomes apparent during early sequences.

5.2.1 Primary Reactor (R-1) and Process Chemistry Evaluations

Process chemistry and reactor operability will be evaluated throughout the majority of the test program (all but TS-4 and TS-16) through a systematic variation of reactor primary (independent) variables. TS-1 through 12 are concerned with operation utilizing the first type of coal. TS-1 through 8 utilize 100 mesh top-size coal feed while TS-9 and 10 utilize 32 mesh top-size coal feed. Sequences 11 and 12 utilize 10 mesh top-size. The data obtained during test runs operating with similar conditions except for coal particle size differences will be utilized to determine particle size effects on process chemistry and mechanical operability of such reactor associated equipment as mixers and oxygen injection systems. During the evaluation of particle size effects in the primary reactor, this same variable will be studied in all other coal handling operations throughout the process such as the secondary reactor, mix-feed tank, filter, centrifuges, solvent stripper, etc.

Test Sequences 1, 2, 3, 5, 6 and 7 evaluate primary reactor (R-1) operation and process chemistry with -100 mesh coal feed. The primary intent during these early operations is to characterize process chemistry when utilizing the -100 mesh feed to optimize reactor operation. TS-1 and 2 utilize the mild leach solution (2% iron), fairly dilute slurry (20% coal) and maximum reactor residence time (coal feed equivalent of 250 lb/hr) to evaluate reaction pressure and temperature effects at these conditions and ensure process control under maximum temperature-pressure conditions. TS-3, utilizing the mild leach solution, evaluates reactor residence time effects on process chemistry as well as reactor operability and control while operating at maximum temperature-pressure conditions. It should be noted that all other processing equipment will be evaluated for residence time effects during this test. TS-5 will evaluate process chemistry and characterize reactor operation as a function of leach solution concentration when the percent iron in solution is increased from 2% to 3.5% and finally to 5% while operating at maximum temperature-pressure conditions and moderate residence times (500 lb/hr coal feed equivalent). TS-6 will then evaluate the effects of residence time and slurry concentration on process chemistry and reactor operation by increasing system feed rates from 500 lb/hr to 1000 lb/hr coal feed equivalent (design capacity of plant) while maintaining strong leach solution, maximum temperature-pressure conditions and then increasing slurry concentration from 20% to 40% coal. If during the course of this test, reaction and regeneration rates appear to be sufficiently high so as to allow for even shorter residence times in the reactor, the reactor feed will be injected into the ten-stage reactor in a downstream stage (such as the third or fifth stage) thus decreasing residence times even further. During the slurry concentration increase, primary variables of all processing equipment handling that slurry will also be evaluated. TS-7 will be utilized to evaluate process chemistry effects and mechanical operability of alternate methods of oxygenation. Sequences 9 and 10 evaluate temperature and pressure effects on process chemistry utilizing -32 mesh coal while TS-11 and 12 evaluate temperature and pressure effects utilizing -10 mesh coal feed while operating with moderate residence times (500 lb/hr coal feed equivalent), moderate slurry concentration (20% coal), and strong leach solution (5% iron).

TS-13, 14, 16, 17, 18, 19 and 20 are designed to evaluate reaction chemistry and reactor operability on a second type of coal. TS-15 will be used to evaluate residence time effects on process chemistry while operating with strong leach solution (5% iron), moderate slurry concentration (20% coal), and maximum reaction temperatures and pressures (265°F and 100 psig). TS-13, 14, 17, 18, 19 and 20 will evaluate the effects of operating temperature and pressure on process chemistry and operability while operating with -100 mesh, -32 mesh and -10 mesh coal feed. During these operations, all other processing equipment will be evaluated with respect to its operability on type 2 coal feed of various particle sizes.

5.2.2 Secondary Reactor (R-2) Evaluation

During TS-1 through 3, 5 through 7, 9 through 15, and 17 through 20, the secondary reactor (R-2) being utilized as a hold tank (true secondary reactor operation), will have all operating and process chemistry parameters monitored and analyzed. During its operation as a holding tank R-2 requires no primary variable (independent variable) testing since its operation is totally a function of primary reactor (R-1) operation. However, TS-8 will be devoted to utilizing the secondary reactor as a scaled-up primary reactor design scalability. During this operation, the effects of oxygen pressure (utilizing pump around oxygenation technique) will be evaluated while operating at design capacity, maximum temperature, moderate slurry concentration and strong leach solution.

5.2.3 Slurry Mix Tank (T-2) Operation

Slurry mix tank (T-2) primary variables will be evaluated during Sequences 1, 6, 9, 11 and 13. It is intended that the operation of this unit be well established early to ensure properly mixed feeds to the reactor (R-1). It will, therefore, be tested during the first sequence. TS-1 and 6 will determine primary variable effects on 20% and 40% coal slurry operation while TS-1, 9 and 11 will determine effects while operating with -100, -32 and -10 mesh coal feeds at moderate (20%) coal slurry conditions.

6.2.4 Thickener (R-3) and Hydroclone (SP-2) Operations

Since thickener and/or hydroclone operations represent economically attractive alternates or supplements to filtration (especially for the 20% coal slurries) their primary variable effects will be carefully evaluated during TS-8, 10, 12, 13, 18 and 20. During these sequences, the primary variables will be independently evaluated during operation on -100, -32 and -10 mesh type 1 and type 2 coals present in a 20% coal slurry feed.

6.2.5 Evaporator-Crystallizer (SP-3) Operation

TS-4 and 16 are devoted solely to the evaluation of the evaporator-crystallizer in off-line operation. The feed to the crystallizer will be obtained from either of two waste storage tanks (T-25A or T-25B). During TS-4, the feed will be reactor effluent leach solution generated during TS-3, which operated with -100 mesh type 1 coal feed in a 20% slurry 2% iron content leach solution at 265°F and oxygen pressure at 100 psig. The primary reactor variable evaluated during TS-3 will have been coal feed rate (i.e., residence time). During TS-16, the feed will be reactor effluent leach solution generated during TS-15, which operated with -100 mesh type 2 coal in a 20% slurry with 5% iron content leach solution at 265°F and oxygen pressure at 100 psig. Again, the primary reactor variable evaluated during TS-15 will have been coal feed rate (i.e., residence time).

Crystalline product (formed during independent primary variable testing) recovery from the evaporator-crystallizer will be evaluated utilizing both centrifuge and filtration techniques. The evaporator-crystallizer operation will be further evaluated, if this need becomes apparent during operation, by inserting additional test sequences involving this operation during "off weeks".

6.2.6 Filter (S-2, S-3, S-4) Operation

Filter primary variables will be evaluated during TS-1, 2, 3, 5, 6, 9, 11, 14 and 17. Primary variable effects will be evaluated during TS-1 to ensure acceptable (not necessarily optimum) filter operation during following test sequences. During Sequences 5 and 6, filter operation on two slurry concentrations (20% and 40% coal) will be characterized. A comparative evaluation of the operability of each of the three filter types, while operating in similar service, will be performed during TS-1, 2 and 3. This will be accomplished by utilizing each of the three filters as the lead unit during the three sequences (a different filter during each sequence). The filters will then be ordered in the most efficient way (as so indicated by data obtained during TS-1, 2 and 3) for the remainder of the test program.
During TS-5, 9 and 11, filter operation with -100, -32 and -10 mesh type 1 coal will be evaluated. During TS-5, the effect of iron concentration in the leach solution will also be determined, as will be the effect of washing with leach solution contaminated wash water. During TS-14 and 17, filter operability on -100 and -32 mesh type 2 coal will be examined. In addition to the above mentioned testing, the belt filter will also be evaluated with respect to its ability to countercurrently wash coal during filtration during one test of each of the coal types. This test will be performed when schedules and operator availability permit. Also, during evaluation of each of the two drum filters in the last filtration position (prior to the azeotrope still), steam drying of the filter cake prior to discharge from the drum will be evaluated.

6.2.7 Wash Water Contactor (T-11, T-13) Operation

Wash water contactor primary variables will be independently evaluated during TS-2, 9 and 11. During these evaluations, the effects of operating with -100, -32 and -10 mesh coal sizes will be determined with respect to mixer power requirements and coal cake to wash water ratio requirements.

5.2.8 Azeotrope Still (T-14)

Azeotrope still operation will be independently evaluated during TS-3, 10, 12 and 15. During TS-3 and 15, the operation will be characterized as a function of coal throughput (500 lb/hr and 1000 lb/hr) utilizing each of the two coal types at -100 mesh particle size. Sequences 3, 10 and 12 will evaluate operation of -100, -32, and -10 mesh coal at a throughput of 500 lb/hr.

5.2.9 Solvent Centrifuge (S-5, S-6) Operation

Solvent centrifuge variables will be evaluated independently during TS-3, 9, 11, 14, 17 and 19. Centrifuge variables will be characterized as a function of throughput during Sequence 3 utilizing -100 mesh coal. Centrifuge variables will also be evaluated for -100, -32 and -10 mesh coal slurry operation with both the type 1 and type 2 coals. These determinations will be obtained during TS-3, 9 and 11 for the type 1 coal and TS-14, 17 and 19 for the type 2 coal.

5.2.10 Solvent Contactor (T-16) Operation

Solvent contactor primary variables will be evaluated as a function of coal feed particle size for the type 1 coal. Test sequences to be utilized are TS-2 (-100 mesh coal), TS-9 (-32 mesh coal) and TS-11 (-10 mesh coal).

5.2.11 Solvent Stripper (SP-4) and Coal Cooler (E-4) Operation

Solvent stripper and coal cooler primary variables will be independently tested during TS-5, 10, 12, 13, 19 and 20. Variables will be evaluated as a function of coal type and particle size. Sequences 5, 10 and 12 will utilize -100, -32 and -10 mesh type 1 coal, while Sequences 13 and 19 will utilize -100 and -10 mesh type 2 coal. TS-20 will utilize steam instead of nitrogen as the stripping media. In the steam stripping operation (TS-20) the coal feed is -10 mesh type 2 coal.

6.2.12 Solvent Cooler (E-9) Operation

The solvent cooler will be independently evaluated during TS-6 as a function of feed rate (solvent residence time).

6.2.13 Sulfur Filter (S-7, S-8) Operation

Sulfur filter primary variables will be evaluated independently during TS-2 as a function of toluene sulfur content.

6.2.14 Pulverizer System (A-3), Knock-Out Drum (V-1), Cooling Water Drum (T-5), Barometric Condenser (E-1,2,7), Vacuum Pumps (K-2,3,4), Solvent Still (C-1), and Carbon Absorption Drum (SP-5) Operations

The operations of these pieces of equipment will not involve any independent primary variable testing; however, all dependent secondary variables will be monitored throughout every test sequence to characterize equipment operation as a function of all process operating conditions studied.

6.3 MATERIALS TESTING

In addition to the above described equipment operations testing, a materials testing program will be conducted throughout the entire 9-month test schedule. The test program will involve the evaluation of test samples of various materials of construction. These various test samples will be subjected to the most chemically active and corrosive environment in the process; namely, the elevated temperature (to 265°F), highly oxidative (to 100 psig dispersed oxygen), strongly acidic (pH as low as 1), highly reactive (ferric/ferrous sulfate), highly erosive (as high as 40% coal slurry) primary reactor environment. Test samples of pipe elbows and straight piping will be fabricated for placement into the slurry-oxygen injection recirculation loops. It is here that the highest slurry flow velocities are obtained and that maximum corrosion and/or erosion is to be expected. The elbows should be most susceptible to stress corrosion. Also, test coupons of the various candidate materials will be fabricated and mounted on the inner walls of the reactor. These samples will be subjected to the most chemically reactive environment found in the process. Both types of samples will have welded joints so that mechanical and heat induced stress corrosion can be evaluated. Dissimilar materials will also be welded together and put into test service so that galvanic type corrosion can be evaluated.

Material samples will be periodically removed and visually inspected. Sample inspections will take place after every 4th week of process operation (during down-time) unless sample failures indicate the need for more frequent inspections. If significant visual damage is present on the samples, the corroded part (either pipe or coupon) will be replaced with another test specimen of the same or different material. The spent specimen will then be metallurgically evaluated. Special effort will be made to accurately profile the test environment, including both operation and stagnant down time. This effort will yield a large data base of information with respect to materials of construction for this specific service. This data will find direct application to full-scale process evaluations.

6. REACTOR TEST UNIT

The complete and highly automated pilot plant for evaluating the Meyers Process (described in Section 3.0 of this report) includes coal grinding, leach/regeneration of fine coal slurry, washing for sulfate removal, solvent extraction for elemental sulfur removal, drying for solvent recovery, crystallization for iron sulfate disposal and alternate processing equipment in the coal separation steps. Since flexibility was required for pilot plant testing, an unusual amount of complexity was necessarily introduced into both the reactor section and downstream operations so as to permit an adequate variable test range in the reactor sections. As a consequence, well over 100 items of major equipment, and nearly 1000 instruments and controls were included in the design. Therefore, preliminary design was also prepared for a less complex test plant which could be used to assess key process variables at reduced construction and operating cost. The result of this effort was the development of a dual reactor testing system with capability to evaluate critical processing variables. The design and operation of that less complex plant, The Reactor Test Unit (RTU), is the subject of the following sections of this report. Section 6.1 presents the process design, 6.2 presents unit start-up procedures, and 6.3 discusses unit operation and supplier testing considerations.

6.1 RTU PROCESS DESIGN

The primary objective of the RTU process design effort was to obtain the key reactor/regeneration information on fine to intermediate coal sizes and on a readily shippable coarse coal at a scale where equipment representative of commercial types could be utilized. A secondary objective was to reduce the installed cost of the RTU substantially below that of the complete pilot plant without compromising primary objectives. The resultant RTU design is shown in the process flow diagram which appears in Appendix F. The plot plans and an artist's sketch of the facility are presented in Appendix G.

In the paragraphs which follow, a description of the equipment and the principal factors considered in the process design are presented. A detailed reactor test unit equipment listing is presented in Appendix H.

6.1.1 Fine Coal Feed System

Coal ground to a top size of 8 mesh or less can be handled by the fine coal processing train (Appendix F). The coal is received in commercially available tote bins of 2 ton capacity (A-1) filled to a small positive pressure with inert gas to minimize coal weathering. The fine coal storage tank (T-1) is equipped at the top with a tote bin tipping and unloading mechanism available from the tote bin supplier. The tote bins are lifted and positioned in the unloader by a small electric motor driven crane. The tank is sized for about 1-1/2 tote bin loads to allow for continuous operation, although a single bin (4000 lbs) will provide 2 shifts of operation at the nominal 250 lb/hr coal feed rate.

The storage tank is discharged (A-2) to the weigh belt (A-3). The weigh belt which controls the coal feed rate, feeds the coal through the rotary feed valve (SP-1) into the mix tank (T-2). Nitrogen purge and a pressure control relief valve provide an inert atmosphere in the storage tank and nitrogen purge of the rotary feeder prevents steam in the mix tank from entering the dry coal feed system. A sliding door and a hopper on the mix tank feed pipe allows solids, such as makeup iron sulfate, to be added to the system.

6.1.2 Fine Coal Wetting

A three-stage mix tank (T-2) equipped with mixers (M-1 to M-3) provide for slurrying and wetting the dry coal. Warm leach solution from the scrubber mist eliminator (SP-2) is introduced into the first stage. At nominal feed rates (250 lb/hr coal and 500 lb/hr leach solution), the residence time is 15 min per stage when the tank is three-fourths full. The slurry cascades from stage to stage through adjustable baffles that adjust the residence time. Complete mixing is provided in each stage and the baffles prevent return of slurry to an upstream stage. All liberated gases and the nitrogen purge gas are removed by a blower (B-1) after first scrubbing in SP-2 with incoming leach liquor to remove foam, and finally with plant cooling water in the cooling water drum (T-3) to remove traces of entrained material and visible water vapor.

6.1.3 Fine Coal Primary Reactor

The primary reactor (R-1) is designed for a hold-up of 5 hours operating 80 to 90% full at the nominal feed rate. Slurry at pressures up to 125 psig is fed by a diaphragm-type slurry pump (P-1). The reactor design pressure and temperature are 125 psig and 275°F with nominal operating conditions of 50 psig and 250°F.

The interior of the cylindrical reactor vessel is divided into ten compartments, each with a length approximately equal to a slurry depth. Baffles are provided to allow for cascade flow from stage to stage and to prevent return of slurry to an upstream stage. Continuous regeneration of the leach solution in each of the first five compartments or stages is carried out by injection of oxygen into the discharge line of a slurry recirculation pump connected to each stage (P-2 to P-6). Each stage is provided with an agitator (M-4 to M-13) with an impeller installed near the vapor-liquid interface in order to reduce the possibility of any significant build-up of solids in the vapor space.

The agitators also may be provided with a second submerged impeller suitable for gas dispersion so as to demonstrate mechanical aeration as a means of leach solution regeneration. The gas dispersion agitators can be installed in any compartment. A manifold system equipped with static mixers is installed in each of the last five stages to provide for demonstration of a third means of leach solution regeneration.

Injection connections for steam and for cool recycle leach liquor are provided for temperature control. The last five compartments can be bypassed and slurry withdrawn from the fifth compartment in order to operate with only five stages if desired.

Oxygen is obtained from liquid oxygen storage, vaporized and supplied at regulated pressure. The excess oxygen saturated with water vapor at the operating temperature is vented through knock-out drum (V-1) where it is scrubbed and cooled by cool return leach solution. The warmed leach solution containing water condensed from the vent oxygen is fed to the mix tank (T-2) through the scrubber (SP-2). The cooled, scrubbed vent oxygen is reduced to ambient pressure and washed by plant cooling water in T-3 before being vented to the atmosphere.

The flash drum (T-4) is designed to provide a means to disengage the steam flashed after reduction in pressure of the primary reactor effluent to atmospheric pressure. The drum is equipped with a tangential entry and a mesh pad mist separator on the steam discharge line. The steam is condensed and scrubbed with cooling water in T-3 prior to venting any residual gas.

6.1.4 Fine Coal Secondary Reactor

Slurry from the flash drum (T-4) may go directly to filtration or may be charged to the secondary reactor (R-2). The reactor is sized to hold two hours of reactor effluent at nominal flow. It is planned that after filling with slurry the reactor will be held for times up to about 24 hours while the temperature is maintained near the boiling temperature by insulation containing electrical heaters. Coal slurry in the secondary reactor may be stirred, to simulate a flow through reactor, or unstirred, to simulate a thickening tank. It is planned that an acid resistant concrete lining will be applied to the internal of the reactor to explore the potential usefulness of this material for commercial scale process equipment. When the reaction period is complete the secondary reactor will be discharged to the filter through slurry pump P-7. Although the higher pressure rating of P-7 is not required for secondary reactor discharging, the pump selected is identical to the primary reactor feed pump (P-1) and serves as its spare.

6.1.5 Coal Filtration

A belt filter (S-1) has been selected for the separation step since it is applicable to coal of all particle sizes. The skid mounted package filtration unit includes the belt filter, the filtrate receiver (V-2) and pump (P-9), the wash water receiver (V-3) and pump (P-10), and the vacuum pump (K-1). The filter is equipped with filter cake wash sprays for hot water or steam and has provision for filter cloth washing and disposal of the waste wash water by pump (P-13). Normally the filtrate from V-2 will be pumped to the leach solution tank (T-6) containing solution with high Fe^{+3}/Fe ratio (Y).* The wash water from V-3, which contains some residual leach solution, will be pumped to the waste tank (T-7). In those tests where the slurry has a low Y (such as slurry from the secondary reactor (R-2) or the coarse coal reactor (R-3) effluent), filtrate from V-2 will be pumped to leach solution tank (T-5) containing low Y solution. Wet filter cake will be discharged from the filter into covered dumpsters for disposal or be retained for subsequent testing or evaluation.

6.1.6 Coarse Coal Reactor

The coarse coal reactor (R-3) will contain approximately 4000 lbs of coal from tote bins (A-4) identical to those used in the fine coal feed system and will use the same type of tipping and discharging equipment. The reactor will be equipped with insulation containing electrical heaters to maintain reactor temperature at the desired level. Coarse coal leaching will be a batch operation and will include the following steps:

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* As used in the process description, high Y solution contains about 5% iron with 90% Fe^{+3} and 10% Fe^{+2} (Y = .9) while low Y solutions will have an Fe^{+3}/total Fe ratio of about 0.75.

(1) One tote bin of coarse coal (about 2 tons) will be placed in the reactor.

(2) Steam will be purged through the coal until the desired coal bed temperature is reached.

(3) Leach solution (normally high Y solution from T-6) will be pumped at high rate (P-12) through heat exchanger E-1 where it will be heated to the reaction temperature before entering R-3.

(4) Reactor R-3 will be filled with solution (nominally 15 to 30 minutes) while exhaust gases removed by blower B-2 are scrubbed in T-3 before venting.

(5) When the reactor is full, the leach solution feed rate will be reduced to the exchange flow rate (typically less than 1 gpm) with overflow solution returning to the low Y solution tank (T-5).

(6) Solution circulation will be continued for the desired time (nominally 25 to 100 hrs) with solution temperature controlled by exchanger E-1 and reactor temperature maintained by controlling the reactor heaters.

(7) When the planned reaction time is reached, the reactor will be drained of leach solution by pump P-8 and coal samples drawn from several levels and radial positions in the reactor.

(8) The wet reactor contents may be discharged through A-6 to a conveyor (A-5) and washed on the filter (S-1) or may be conveyed directly to a dumpster.

(9) As an alternate to Step 8, the coal may be washed in place by pumping (P-15) water from T-8 through exchange E-1. The overflow wash water will be taken to waste tank T-7.

(10) The washed coal can then be drained, sampled and discharged as in Steps 7 and 8.

Leaching and washing of coarse coal is believed to be an important aspect of the reactor testing program. At present there is insufficient design data to address two anticipated design problems. The height to cross section ratio and requirements, if any, for internal flow distributors are both unknown. Special provisions for reactor discharging to prevent bridging also may be required. Presently, it is thought that a reactor

3' x 3' x 9' high is a likely design, but a longer reactor 2-1/2' x 2-1/2' x 13-1/2' high is shown in the initial design. The support structure planned for the taller reactor can readily accommodate a shorter reactor but the reverse situation would lead to difficulties.

6.1.7 Primary Reactor as a Regenerator

The RTU design allows for the primary reactor (R-1) to serve as a regenerator for leach solution. Solution with low Y from tank T-5 may be pumped (P-11 or P-12) to the reactor at rates up to 20 gpm for separate examination of the regeneration rates. Leaching of each batch of coarse coal will produce up to 10,000 gal. of low Y solution so that up to about 10 hours of continuous regeneration testing can be accommodated even at near maximum rates. It should be noted that normally the low Y solution will be regenerated during fine coal processing rather than by separate regeneration. The test plan will arrange the tests so that solution of the correct Y is available without unnecessary solution adjustments. About two or three test weeks of fine coal processing per batch of coarse coal will keep the solution in balance. Solution tanks sized for 15,000 gal. provide an adequate margin for off-nominal operation conditions.

6.1.8 Sizing the Reactor Testing System

In the preceding paragraphs, the discussion indicated that the initial process design was based on nominal throughputs of 250 lb/hr of fine coal (continuous operation) and 4000 lb/batch of coarse coal. These sizes provide for a test program which balances fine and coarse coal testing. Coal of appropriate size is obtained and handled in 2-ton tote bins which are the largest standard size available commercially. A single bin is a convenient coarse coal reactor batch and one bin also provides for two shifts of operation of the fine coal system. The total quantity of coal to be processed is estimated to be about 150 tons divided between cleaned coal and run-of-the mine coal. This quantity, while large, is reasonable to transport from mine to grinding to use in bin quantities.

The process equipment, however, has a wide range of coal throughput. At nominal slurry concentration and coal feed rate, the flow rate through the fine coal system is about 1.2 gpm. Pump capacity would allow at least a threefold increase to 750 lb/hr and may be capable of reaching 1000 lb/hr. At 20% slurry coal feed rates from 100 to 500 lb/hr should be available. It is believed that the belt filter will handle any of these feed rates except possibly the highest rates with a -100 mesh high ash uncleaned coal. Heating, cooling and storage capacities have been selected to provide for the high testing flexibility felt to be necessary.

6.2 RTU START-UP

The initial start-up, short-term operation and shutdown of each of the two reactor trains will be performed and safety procedures will be practiced. The primary goal of these operations is to check out equipment, personnel, standard and emergency procedures, analytical techniques and overall operability of the integrated systems. The initial start-ups will require elongated time schedules to allow for greater process control and more in-depth procedure and equipment evaluations than will be required during subsequent start-ups. A typical start-up test sequence that would be appropriate for this type of operation is presented in Table 5. It is anticipated that unit start-up operations will immediately precede the RTU testing program and require approximately 2 months to complete.

The philosophy of the start-up operation, as outlined in Table 5, is to initially check out mechanical operation and structural integrity of both the fine and coarse coal treating systems while operating under conditions which vary from very mild to quite severe (i.e., initial water circulation testing at ambient temperature and pressure to final start-up testing utilizing strongly acidic coal slurry at elevated temperatures and pressures). It is also intended that during the course of start-up operations, all process monitoring, data gathering, physical sampling and chemical analysis techniques will be evaluated with acceptable techniques being demonstrated

TABLE 5. GENERALIZED START-UP SEQUENCE

	System	Operation	Objective
1	Fine Coal Reactor System	Process water circulation at ambient temperature to nominal expected operating temperatures. No coal, iron sulfate or oxygen present.	Check integrated operability of pressure, temperature, flow, and level controllers, valves, recorders, indicators, cooling and wash water circuits, process steam, and flash system, etc.
2	Fine Coal Reactor System	Flow oxygen at ambient to 50 psig through [illegible] and [illegible] water injection system.	Check operation and integrity of oxygen delivery system, monitoring, regulators, control equipment, etc.
3	Coarse Coal Reactor System	Process water circulation at ambient to normal boiling point temperatures. No coal or iron sulfate present. Drain reactor.	Check operability of pressure, temperature, flow and level controllers, valves, recorders, indicators, cooling and wash water systems, process steam, and flash system, etc.
4	Fine Coal Reactor System	Drain process water from entire system and refill with purchased distilled water.	Remove any debris (dirt, dust, iron filings, etc.) picked up during circulation operations and also make any required adjustments, repairs, or modifications to the unit.
5	Fine Coal Reactor System	Charge fine coal (~100 mesh) to the storage hopper. Feed reactor and operate system at 20-30% slurry with oxygen injection at 50 psig and 250°F reactor temperature. (No iron sulfate added, distilled water only.)	Check operability of total Fine Coal Reactor System, including filter and washing operations, during slurry operation. Collect samples and operational data to be utilized as reference data (no iron sulfate in leach solution, water only).
6	Fine Coal Reactor System	Charge coarser fine coal (~8 to ~12 mesh). Feed reactor and operate system at 20-30% slurry with oxygen injection and 250°F reactor temperature. (No iron sulfate added, distilled water only.)	Check operability of total Fine Coal Reactor System, including filter and washing operations, during slurry operation. Collect samples and operational data to be utilized as reference data (no iron sulfate in recycle leach solution, water only).
7	Coarse Coal Reactor System	Charge Coarse Coal (~1/4 inch) Reactor System and operate utilizing hot (200°F) distilled water circulation operation, batch wash coal charge to silo and discharge coal utilizing filtration and wet system.	Check operability of total Coarse Coal Reactor System during operation (especially liquid feed rate controllability); collect operational data and samples to be analyzed and utilized as reference data (no iron sulfate in leach solution, water only).

TABLE 5 (Continued). GENERALIZED START-UP SEQUENCE

	System	Operation	Objective
8	Flow Coal Reactor System	Make up leach solution to approximately 2-3% total Fe by feeding iron sulfate crystals into the mix tank (T-2) utilizing the pulverized coal feed [illegible] while circulating hot distilled water [illegible] coal feed.	Check operability of system (especially all rotary equipment, coal flash system, etc.) and insure system integrity during operation with hot strongly acid leach solution.
9	Gravity Coal Reactor System	Circulate hot leach solution (2-3% Fe) through system.	Check operability and integrity of the system while operating under hot strongly acid conditions.
10	Gravity Coal Reactor System	Perform first batch test run. Charge ~[illegible] coal and circulate hot ([illegible]°F) leach solution (2-3% Fe) for two to four days. Upon run completion, grab sample the reacted coal, back wash the coal in situ, grab sample the washed coal and discharge utilizing the filter.	Demonstrate total Gravity Coal Reactor System operability, demonstrate adequacy of sampling and analytic techniques and generate a complete set of process evaluation data.
11	Flow Coal Reactor System	Perform first total test run. Charge with ~[illegible] coal, operate with 2-3% Fe leach solution, 20-30% (wt.) coal slurry, oxygen injection pressure of [illegible] psig and a reactor temperature of [illegible]°F. During operation (over an approximate [illegible] hour period) sample all appropriate processing streams including washing and filtration operations.	Demonstrate total Flow Coal Reactor System operability, demonstrate adequacy of sampling and analytic techniques and generate a complete set of process evaluation data.

during final start-up runs. Additionally, it is during start-up testing that reference data using pure water feed simulating the iron sulfate leach solution will be generated.

6.3 REACTOR TEST UNIT OPERATION AND EQUIPMENT SUPPLIER TESTING

Following start-up operations, the reactor systems (both coarse and fine coal) will be put into operation to characterize process chemistry and equipment operability on two classes of one type of coal (cleaned and uncleaned). The purpose of the test program will be to operate the reactor system to characterize mixing operations, reaction, and regeneration operations and to evaluate initial filtration parameters. Another objective of the test program will be that of generating sufficient quantities of reacted coal product to be sent to equipment manufacturer testing facilities for pilot scale testing of downstream process equipment (filtration, centrifugation, solvent stripping, steam stripping, drying, crystallization, etc.).

6.3.1 Task 3-A, Reactor System Test Operation

The following paragraphs will describe the anticipated approach to meeting the above mentioned objectives for each major section of the reactor test facility and present a possible generalized test plan for utilization during process evaluation. It is anticipated that the reactor testing program will require approximately 7 months to complete.

6.3.1.1 Fine Coal Reaction System - The fine coal reaction system is comprised of all equipment with the exception of six items specific to coarse coal processing, namely the coarse coal blower (B-2), the coarse coal reactor-washer (R-3), the portable conveyor belt (A-5), the coarse coal heat exchanger (E-1), the wash water storage tank (T-8), and the water transfer pump (P-15). For purposes of discussion, the fine coal reactor system (all other equipment) will be further subdivided into the following functional sections: the feed/mixing section, the reaction section, and the filtration section.

Feed/Mixing Section

The primary functions of this section are to deliver pre-ground coal to the fine coal storage tank, sufficiently mix the pulverized coal and leach solution to ensure wetting and defoaming, and to deliver the slurried coal to the reactor. The equipment which constitutes the feed/mixing section is listed below:

Feed Bin (A-1)
Bin Discharger (A-2)
Weigh Belt (A-3)
Rotary Feeder (SP-1)
Storage Tank (T-1)
Feed Mix Tank (T-2)
Feed Tank Mixers (M-1, 2, 3)
Scrubber Blower (B-1)
Scrubber (SP-2)
Cooling Water Drum (T-3)
Feed Pump (P-1)

Anticipating that mechanical operability of all equipment will have been previously demonstrated during plant start-up, the only equipment in this section requiring operational characterization is the slurry feed-mix tank (T-2) and the scrubber-mist eliminator (SP-2). The parameters to be evaluated during slurry feed-mix tank operation are (1) coal characteristics, (2) (2) coal particle size, (3) slurry residence time (15 minutes to 1 hour) which will be varied through the use of liquid level control, (4) slurry temperature (180°F-215°F) which will be varied by steam injection; (5) iron sulfate concentration, and (6) slurry concentration.

Sampling requirements for characterization of this operation include (1) pulverized coal feed sampling to determine particle size distribution, (2) coal slurry sampling from the feed-mix tank effluent which will be analyzed for pyrite conversion and examined to determine degree of wetting and general slurry composition, and (3) gas sampling from gas effluent lines running from the feed mix tank to the scrubber-mist eliminator (SP-2) which will be analyzed for carbon dioxide content (carbon dioxide must be evolved prior to entrance into the reactor section in order to minimize full scale oxygen bleed requirements) and foam content. In addition, the effluent from the scrubber-mist eliminator (SP-2) will be monitored for the presence of foam and acid mists and both the gaseous effluent and aqueous effluent from the cooling water drum (T-3) will be sampled and analyzed for particulate and sulfate presence.

Reaction Section

In this section of the system the bulk of the pyrite contained within the coal is converted to elemental sulfur and soluble iron sulfate and regeneration of the spent leach solution takes place. This section of the process consists of the following group of equipment:

Primary Reactor (R-1)
Reactor Mixers (M-4 through 13)
Knock-Out Drum (V-1)
Flash Drum (T-4)
Secondary Reactor (R-2)
Secondary Reactor Mixer (M-14)
Reactor Circulation Pumps (P-2 through 6)
Secondary Reactor Discharge Pump (P-7)
Leach Solution Surge Tanks (T-5, 6)
Leach Solution Circulation Pump (P-11)
Leach Solution Feed Pump (P-12)

The equipment in this section requiring operational characterization are the primary and secondary reactors (R-1, 2). The primary reactor (R-1) will be characterized in a steady state operating mode while serving as a simultaneous reactor-regenerator. The secondary reactor (R-2) will be characterized while operating in a stirred batch mode, essentially simulating a holding tank, and also unstirred, simulating a thickener. During this operation, no regeneration will take place.

Operational variables to be evaluated during reactor operation include (1) coal characteristics, (2) coal particle size, (3) slurry residence time (1 to 10 hours), controlled through slurry feed pump (P-1) pumping rate and number of reactor stages utilized, (4) reaction temperature (205°F to 265°F), controlled by varying inlet slurry temperature and steam injection, (5) oxygen partial pressure and flow rates, varied through changes in oxygen supply pressure and inlet flow rate adjustment, (6) mode of three phase (oxygen-coal-leach solution) mixing, which will be evaluated utilizing oxygen injection in slurry pump-around loops, static mixing, and oxygen injected through simple agitation, (7) slurry concentration (coal/leach solution ratio), and (8) salt concentration.

Sampling requirements in this section of the process will include slurry samples drawn from each stage of the primary reactor and samples drawn from the secondary reactor effluent. The solid/liquid ratio will be determined and the coal will be separated and analyzed for pyrite and elemental sulfur to determine degree of pyrite conversion to sulfur and sulfate. The leach solution will also be analyzed for ferric and ferrous ion to determine degree of regeneration. The reactor (R-1) liquid effluent (slurry) will also be analyzed for trace materials to evaluate the problem of impurities buildup in the leach solution due to recycling. Oxygen consumption will be determined by mass balancing around the reactor system. The gaseous effluent from the primary reactor (R-1) will be sampled prior to the knock-out drum (V-1) and analyzed for particulate, acid mist concentration and the presence of foam. The cooling water drum (T-3) effluent will be sampled and analyzed for particulate and acid content.

Filtration Section

In this section of the process the slurry leaving the reactors is separated, leach solution collected, and the soluble sulfate washed from the coal matrix and removed from the system as by-product. This section consists of the following equipment:

Filter (S-1)
Filtrate Receiver (V-2)
Wash Water Receiver (V-3)
Waste Disposal Tank (T-7)
Waste Disposal Pump (P-14)
Filter Waste Pump (P-13)
Filtrate Pump (P-9)
Wash Water Pump (P-10)
Vacuum Pump (K-1)

The only equipment in this section requiring thorough characterization is the filter (S-1). The remainder of the equipment will not require characterization since its mechanical operability will have been previously demonstrated during start-up.

Filtration and wash efficiencies of the belt type unit will be determined as a function of (1) input slurry concentration and temperature, (2) coal characteristics and particle size, (3) belt speed (i.e., cake thickness), (4) wash water feed rate, and (5) suction pressure.

In general, all filter influents and effluents will require sampling and analysis for complete characterization. The slurry feed, filtrate, spent wash solution and filter cake will be analyzed for iron sulfate to evaluate wash and filtration efficiencies and determine the fate of the soluble salts. Additionally, the moisture content of the cake will be determined. Also, the effluent from the vacuum pump (K-1) will be sampled and analyzed periodically for particulate sulfate presence for evaluation of possible adverse environmental impact.

6.3.1.2 Coarse Coal Reactor System - The coarse coal reactor system is comprised of three functional sections, the Coarse Coal Reactor-Washer Section, the Leach Solution Regeneration Section, and the Filtration Section.

Coarse Coal Reactor-Washer Section

The primary functions of this section are to demonstrate pyritic sulfur conversion to elemental sulfur and soluble sulfate in large particle size (as large as -3/8 inch) coal and to evaluate in situ washing of the reacted coal to remove the soluble sulfate. The equipment comprising this section is listed below:

Coarse Coal Reactor-Washer (R-3)
Coarse Coal Feed Bin (A-4)
Coarse Coal Bin Discharger (A-6)
Coarse Coal Blower (B-2)
Cooling Water Drum (T-3)
Heat Exchanger (E-1)
Leach Solution Surge Tanks (T-5, 6)
Leach Solution Circulation Pump (P-11)
Leach Solution Feed Pump (P-12)
Water Storage Tank (T-8)
Water Transfer Pump (P-15)
Wash Water Return Pump (P-8)
Portable Conveyor Belt (A-5)

Of the equipment in this section, only the coarse coal reactor-washer (R-3) requires thorough characterization. Operational variables will be examined while the reactor is operating as a fixed-bed reactor and also when it is operating as a fixed-bed coal washer. Operational variables to be evaluated include (1) coal characteristics, (2) coal particle size, (3) coal residence time (1 to 7 days), (4) reaction temperature (to 215°F), (5) inlet and outlet leach solution iron (Fe^{+2} and Fe^{+3}) concentrations, (6) leach solution circulation rate, (7) leach solution salt concentration, (8) wash water circulation rates, and (9) wash water temperature.

Sampling requirements in this section will include coal samples withdrawn periodically (radially and at selected heights of the reactor) which will be analyzed for pyrite, sulfate and elemental sulfur to determine degree of pyrite conversion (during reaction) and washing efficiency (during washing cycle). The inlet and outlet leach solution will be analyzed for ferric and ferrous ions to determine reaction rates. Wash water exiting the fixed-bed unit will also be sampled and analyzed for iron and sulfate contents so as to determine soluble sulfate extraction rates from coarse coal. Additionally, the gaseous effluent from the cooling water drum (T-3) will be periodically sampled and analyzed for particulate and acid mist content.

Leach Solution Regeneration Section

The Leach Solution Regeneration Section will be utilized to characterize leach solution regeneration operations and to supply regenerated leach solution to the process surge tanks (T-5 and T-6) as required. The equipment which constitutes this section is as follows:

Primary Reactor-Regenerator (R-1)
Reaction Recirculation Pumps (P-2 through 6)
Reactor Mixers (M-4 through 13)
Flash Drum (T-4)
Knock-Out Drum (V-1)
Cooling Water Drum (T-3)

Variables to be evaluated during regeneration studies will include (1) leach solution residence time in R-1, (2) regeneration temperature, (3) oxygen feed rate, (4) oxygen pressure, and (5) mode of oxygen injection. Feed and effluent leach solution samples will be obtained from the regenerator as well as samples from each regenerator stage. They will be analyzed for iron (Fe^{+2} and Fe^{+3}) content so as to determine degree of regeneration.

Filtration Section

The Coarse Coal Filtration Section will be utilized to study filtration and filter washing characteristics of reacted coarse coal. Descriptions of the equipment which comprise this section, as well as the test variables and sampling requirements associated with its operation, are identical to those presented previously in the Fine Coal Reactor System discussion (Section 6.3.1.1).

6.3.2 Equipment Supplier Testing

During the final 4 months of reactor system operation, large quantities (up to several tons) of reactor processed coal (both fine and coarse) and leach solution will be sent to major equipment manufacturers for evaluation in specific types of pilot scale equipment. Operations such as filtration, centrifugation, steam or nitrogen sulfur stripping, organic solvent removal of elemental sulfur, crystallization, and drying will be evaluated. Table 6 presents a summary of information gathered during preliminary discussions with typical equipment manufacturers. Information outlining test facility locations, feed stock requirements, and anticipated test duration is presented. The following paragraphs describe test objectives and sampling requirements associated with each type of test operation.

6.3.2.1 Filtration - Filtration tests will be performed to characterize the operability of different types of equipment (belt, drum, drum-belt) with respect to filtration and washing capability. The parameters to be studied and sampling requirements for each evaluation are identical to those presented earlier (Section 6.3.1.1) during discussion of filtration testing associated with fine coal reactor system operation.

6.3.2.2 Centrifugation - Centrifuge operation will be evaluated from the standpoint of washing and separation efficiency. Variables which might be evaluated include (1) input slurry composition and temperature, (2) coal characteristics and particle size, (3) centrifuge rotational speed, and (4) wash liquor feed rate and temperature. The feed slurry and wash solvent will be analyzed for composition, elemental sulfur and sulfate content. The centrate and product coal cake will be analyzed for coal to liquid ratio as well as sulfur content. These data will indicate the wash and filtration efficiencies of centrifuges in this application.

TABLE 6. SUMMARY OF MANUFACTURER TESTING CAPABILITIES

Operation	Manufacturer	Location of Test Facility	Test Duration[a]	Quantity Required For Testing[a]
Filtration	Ametec	East Moline, Ill.	3-6 Days	5 to 50 gal. slurry[b]
	Denver	Colorado Springs, Colo.	5 Days	2000 to 2500 lb slurry[b]
	Bird Machine Co.	Walpole, Mass.	2-3 Days	500 to 1000 gal. slurry[c]
	Dorr Oliver	Oakland, Calif.	5 Days	30 to 40 gal. slurry[b]
Centrifugation	Ametec	East Moline, Ill.	3-5 Days	5 gal. minimum[b]
	Bird Machine Co.	Walpole, Mass.	5 Days	500 to 1000 gal. slurry[c]
	Dorr Oliver	Oakland, Calif.		1000 gal. slurry[b]
Steam and Solvent Stripping	Denver Equipment	Colorado Springs, Colo.	5-10 Days	Not known
Crystallization	Denver Equipment	Colorado Springs, Colo.	Not known	Not known
	Eimco	Salt Lake City, Utah	Not known	Not known
Drying	Ametec	East Moline, Ill.	Not known	Not known
	Wyssmont	Fort Lee, N.J.	4-5 Days	4000 to 6000 lb wet coal cake

[a] Per coal sample.

[b] Requires coal product and leach solution to be mixed to slurry specification at the test facility.

[c] Must be received premixed in drums.

6.3.2.3 Solvent Extraction and Vapor Stripping - Several techniques for the removal of elemental sulfur (a co-product of the leach reaction) from previously water washed (to remove soluble sulfate) coal may be evaluated. An effective technique used in bench-scale studies involves extraction of washed coal with an organic solvent followed by drying to remove residual solvent. Alternate approaches which have been briefly investigated in the laboratory will be examined and those found to be potentially attractive may be included in the supplier testing.

Variables which might be evaluated include (1) coal characteristics and particle size, (2) stripping temperatures and pressures, (3) feed solvent sulfur content, (4) coal residence time, (5) coal to stripping media ratio, and (6) type of solvent (toluene, xylene, kerosene, etc.). Evaluation of this operation will require that samples of feed and product coal as well as influent and effluent stripping media be analyzed for sulfur content so as to determine overall operation efficiency. Additionally, solvent and/or moisture retention of the product coal will be determined.

6.3.2.4 Crystallization - The crystallizer operations will be evaluated with respect to output product (crystalline iron sulfate salts) characteristics as a function of operational parameters. Variables which might be studied include (1) input leach solution composition, (2) feed rate, (3) operating pressure (sub-atmospheric to one atmosphere), (4) concentrate circulation rates, and (5) heater temperature (i.e., concentrate temperature). Evaluation of this operation will require that concentrate samples be taken and that the crystalline product be analyzed and chemically characterized. Quantities of this by-product will be retained for future study.

6.3.2.5 Drying - Drying operations will be evaluated to determine the efficiency of solvent recovery from coal which has previously undergone organic solvent stripping of elemental sulfur. It is anticipated that inert gas (N_2) and steam circulation techniques will be studied. The characterization of this operation will be determined as a function of (1) input cake composition (i.e., solvent content), (2) coal characteristics and particle size, (3) feed rate, (4) temperature (5) solids residence time, and (6) steam quantity and quality. Cake input and product samples will be analyzed to determine solvent and moisture content in order to evaluate the drying efficiency of the unit. Inlet and outlet temperatures will also be monitored.

7. REFERENCES

1. Hamersma, J. W., E. P. Koutsoukos, M. L. Kraft, R. A. Meyers, G. J. Ogle, and L. J. Van Nice, "Chemical Desulfurization of Coal: Report of Bench-Scale Developments", EHSD 71-7, prepared for the Office of Research and Monitoring of the Environmental Protection Agency, Research Triangle Park, N. C., February 1973.

2. Koutsoukos, E. P., M. L. Kraft, R. A. Orsini, R. A. Meyers, M. J. Santy, and L. J. Van Nice, "Meyers Process Development for Chemical Desulfurization of Coal", Contract No. 68-02-1336, prepared for the Office of Research and Monitoring of the Environmental Protection Agency, Research Triangle Park, N. C., May 1976.

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APPENDIX A

PROCESS FLOW DIAGRAM

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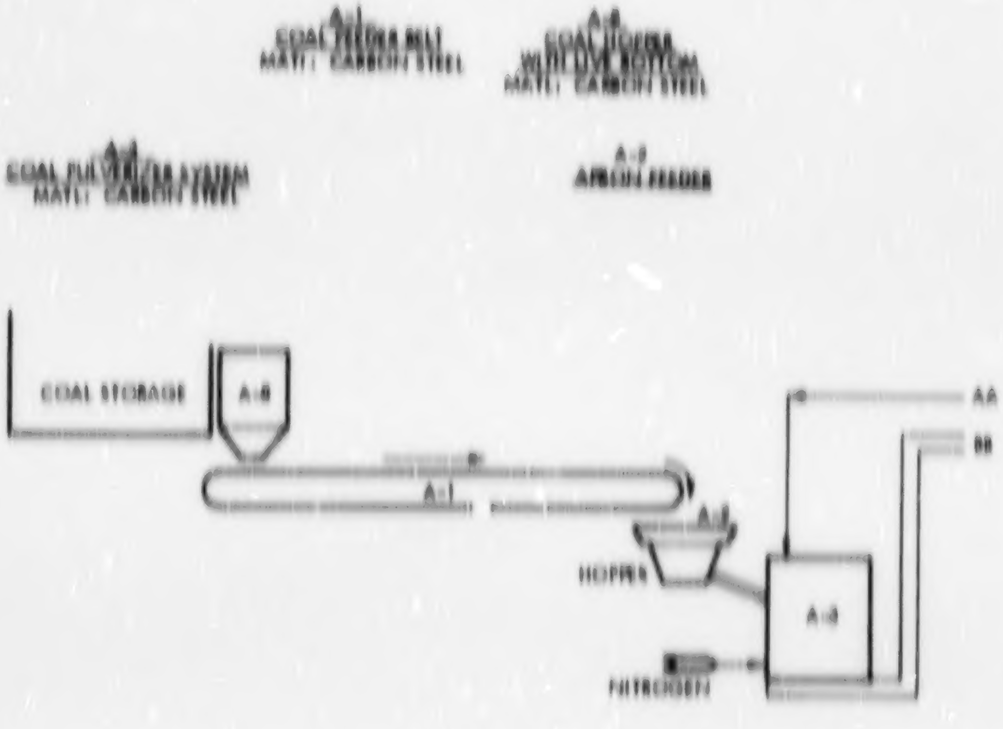
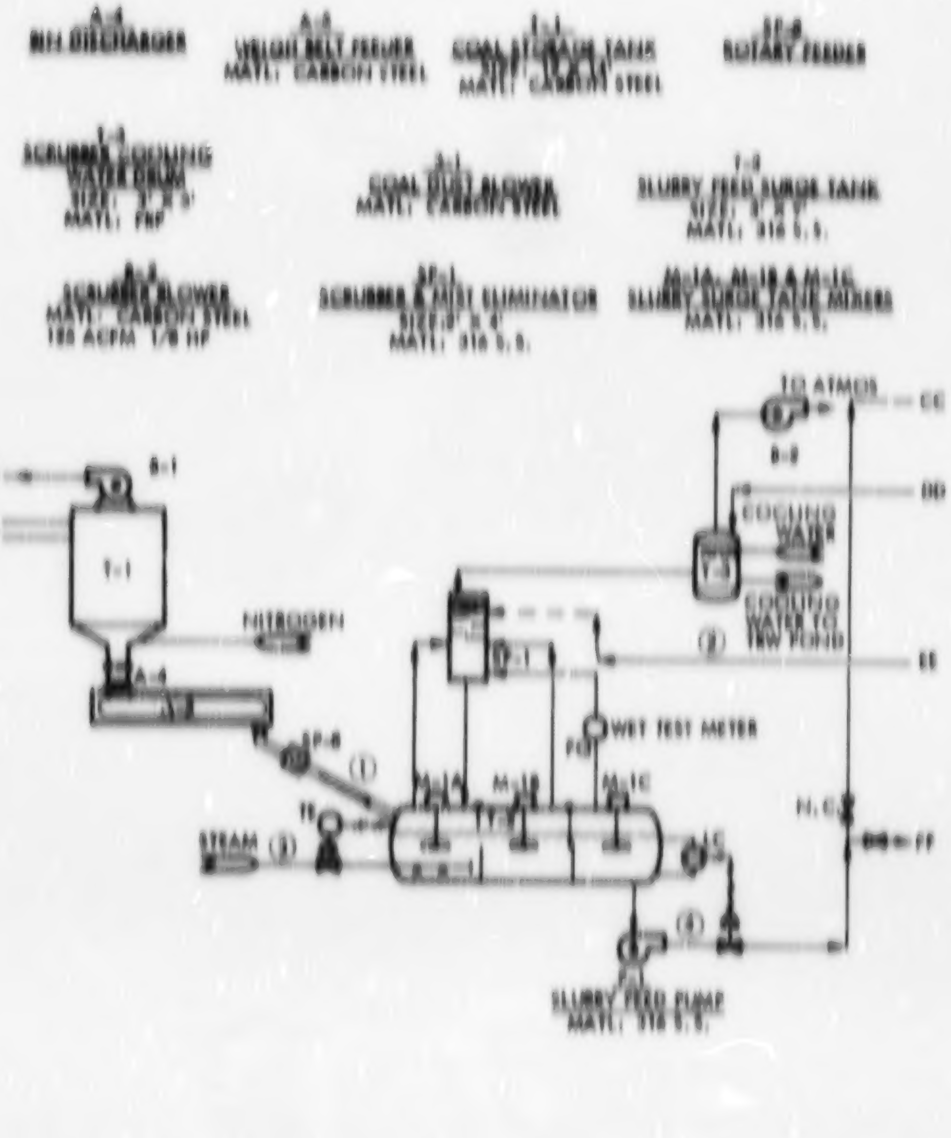
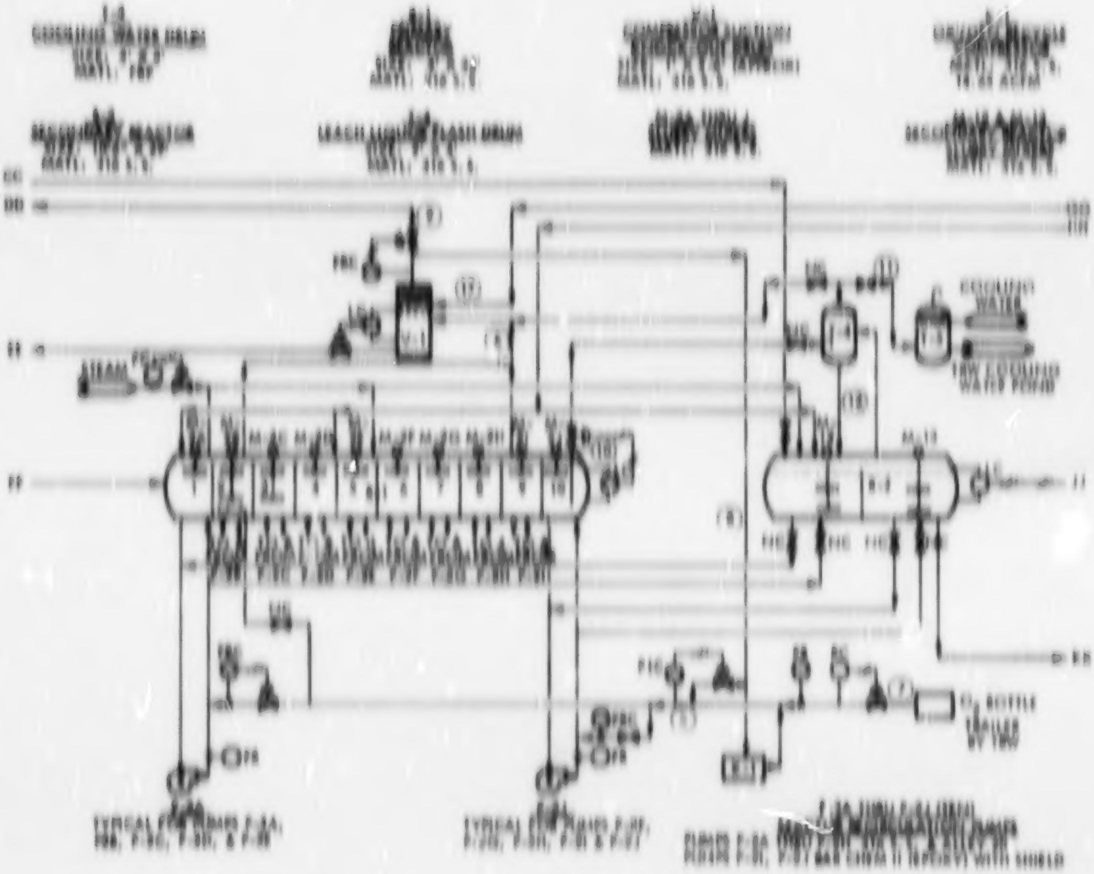
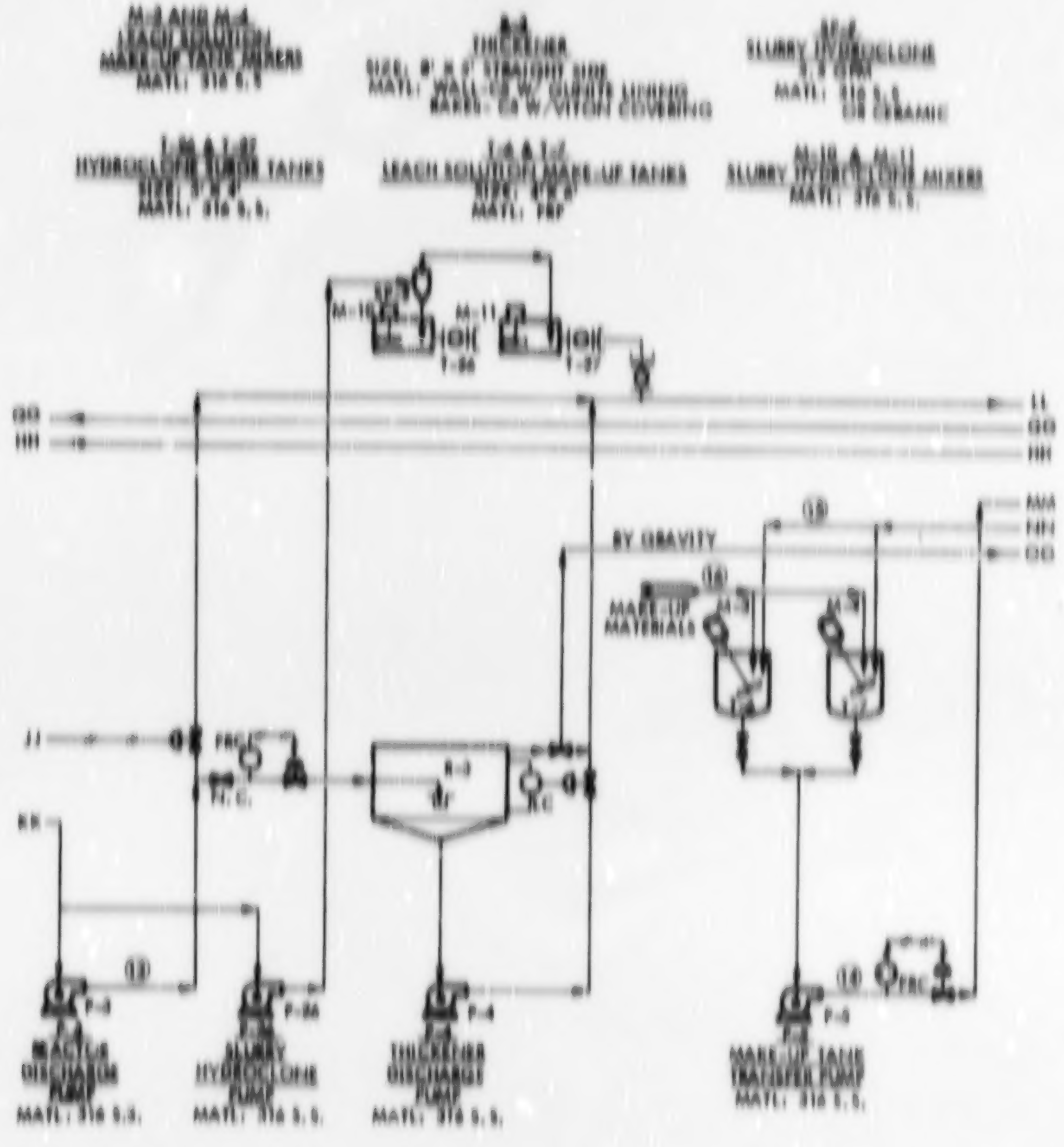
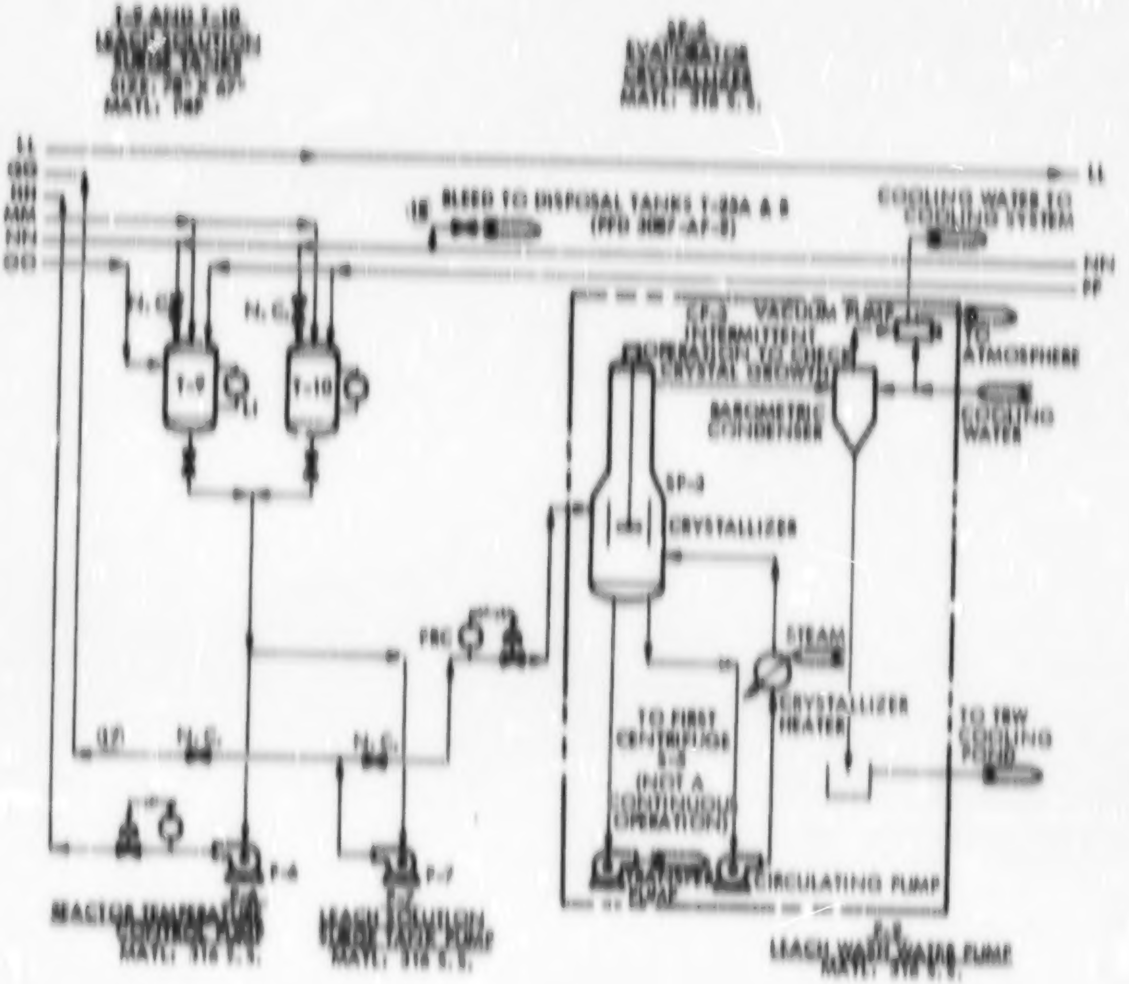
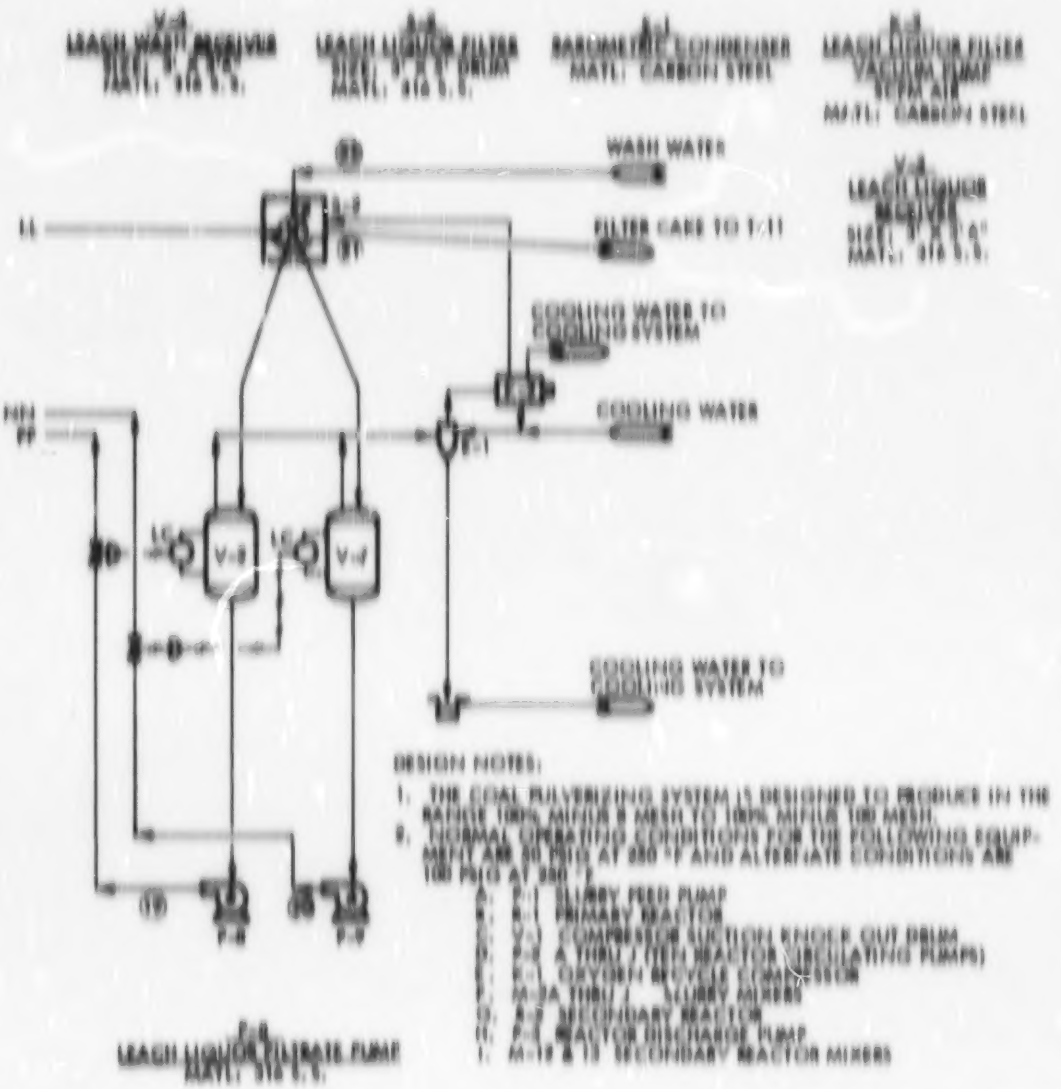

A-1
COAL FEEDER BELT
MAT'L: CARBON STEEL
A-6
COAL HOPPER
WITH LIVE BOTTOM
MAT'L: CARBON STEEL
A-3
COAL PULVERIZER SYSTEM
MAT'L: CARBON STEEL
A-2
APRON FEEDER
COAL STORAGE
A-6
A-1
A-2
HOPPER
A-3
NITROGEN
AA
BB


A-4
BIN DISCHARGER
A-3
WEIGH BELT FEEDER
MAT'L: CARBON STEEL
T-1
COAL STORAGE TANK
MAT'L: CARBON STEEL
RF-6
ROTARY FEEDER
T-3
SCRUBBER COOLING WATER DRUM
MAT'L: FRP
B-1
COAL DUST BLOWER
MAT'L: CARBON STEEL
T-2
SLURRY FEED SURGE TANK
MAT'L: 316 S.S.
B-2
SCRUBBER BLOWER
MAT'L: CARBON STEEL
150 ACFM 1/6 HP
SC-1
SCRUBBER & MIST ELIMINATOR
MAT'L: 316 S.S.
M-1A, M-1B & M-1C
SLURRY SURGE TANK MIXERS
MAT'L: 316 S.S.
TO ATMOS
CC
B-2
DD
COOLING WATER
COOLING WATER TO TRW POND
EE
AA
B-1
BB
T-1
NITROGEN
A-4
A-3
RF-6
(1)
(2)
WET TEST METER
FQ
M-1A
M-1B
M-1C
TI
STEAM
(3)
N.C.
FF
(4)
P-1
SLURRY FEED PUMP
MAT'L: 316 S.S.


CC
DD
FF
STEAM
V-1
(12)
(11)
(13)
(9)
COOLING WATER
1
2
3
4
5
6
7
8
9
10


M-3 AND M-4
LEACH SOLUTION
MAKE-UP TANK MIXERS
MATL: 316 S.S.
S-3
THICKENER
MATL: WALL-CS W/ RUBBER LINING
RAKES- CS W/VITON COVERING
SLURRY HYDROCLONE
MATL: 316 S.S.
OR CERAMIC
HYDROCLONE SURGE TANKS
MATL: 316 S.S.
LEACH SOLUTION MAKE-UP TANKS
MATL: FRP
M-10 & M-11
SLURRY HYDROCLONE MIXERS
MATL: 316 S.S.
M-10
M-11
T-26
T-27
GG
HH
JJ
KK
LL
MM
NN
OO
BY GRAVITY
MAKE-UP MATERIALS
FRC
FRC
S-3
P-3
P-26
P-4
P-5
13
15
16
16
MATL: 316 S.S.
SLURRY HYDROCLONE PUMP
MATL: 316 S.S.
THICKENER DISCHARGE PUMP
MATL: 316 S.S.
MAKE-UP TANK TRANSFER PUMP
MATL: 316 S.S.

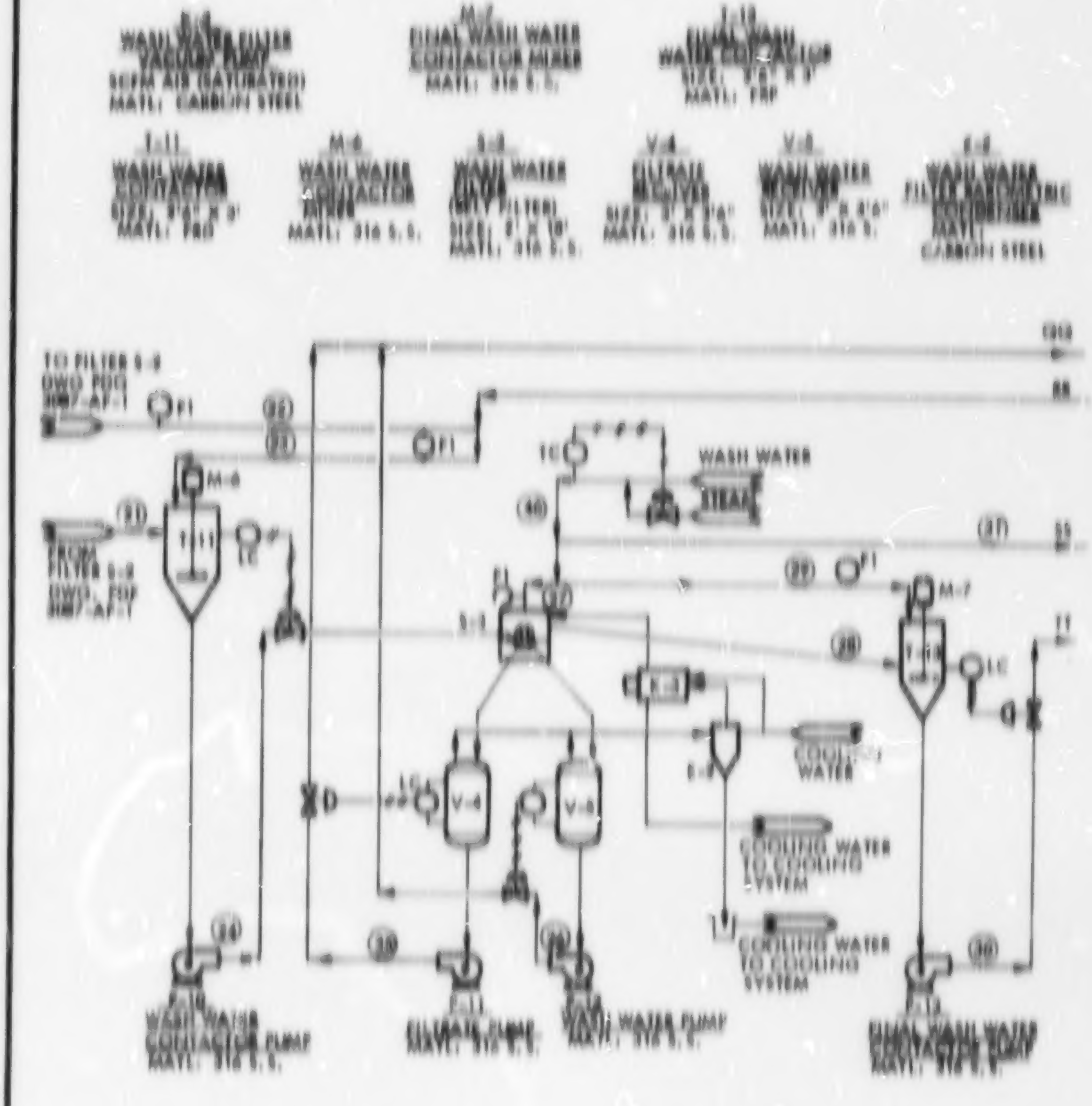
T-9 AND T-10
EVAPORATOR
BLEED TO DISPOSAL TANKS T-20A & B
COOLING WATER TO COOLING SYSTEM
LL
PP
VACUUM PUMP
INTERMITTENT
CRYSTAL GROWTH
TO ATMOSPHERE
BAROMETRIC CONDENSER
COOLING WATER
T-9
T-10
H. C.
EP-2
CRYSTALLIZER
FRC
STEAM
CRYSTALLIZER HEATER
TO FIRST CENTRIFUGE
(NOT A CONTINUOUS OPERATION)
TO TBW COOLING POND
(17)
P-6
P-7
CIRCULATING PUMP
LEACH SOLUTION
LEACH WASTE WATER PUMP

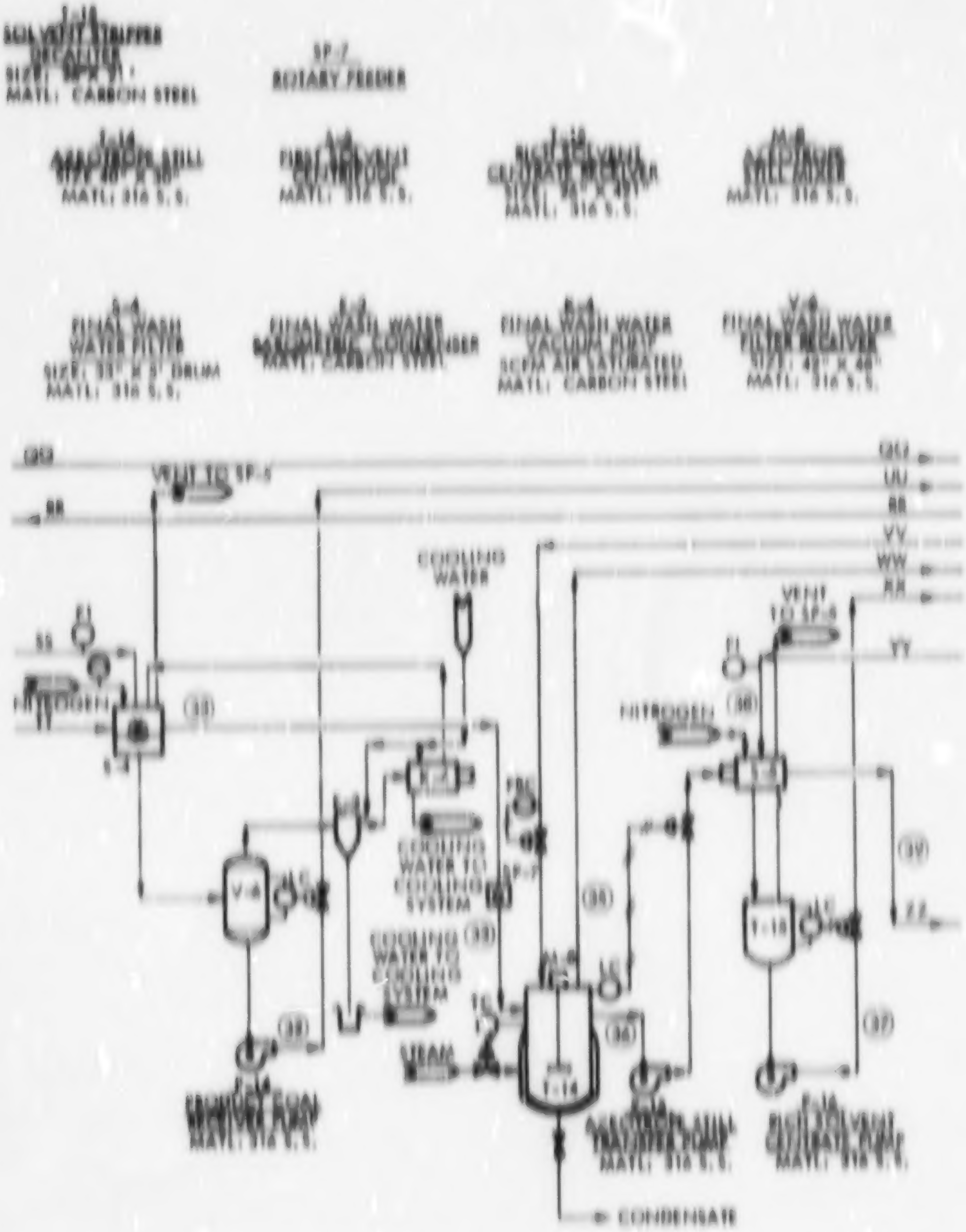

V-4
LEACH WASH RECEIVER
MAT'L: 316 S.S.
F-2
LEACH LIQUOR FILTER
DRUM
MAT'L: 316 S.S.
E-1
BAROMETRIC CONDENSER
MAT'L: CARBON STEEL
K-2
LEACH LIQUOR FILTER
VACUUM PUMP
MAT'L: CARBON STEEL
WASH WATER
V-3
LEACH LIQUOR
RECEIVER
MAT'L: 316 S.S.
LL
F-2
FILTER CAKE TO T-11
COOLING WATER TO
COOLING SYSTEM
COOLING WATER
E-1
HH
FF
V-3
V-4
COOLING WATER TO
COOLING SYSTEM
P-8
P-9
P-8
LEACH LIQUOR FILTRATE PUMP
MAT'L: 316 S.S.

DESIGN NOTES:

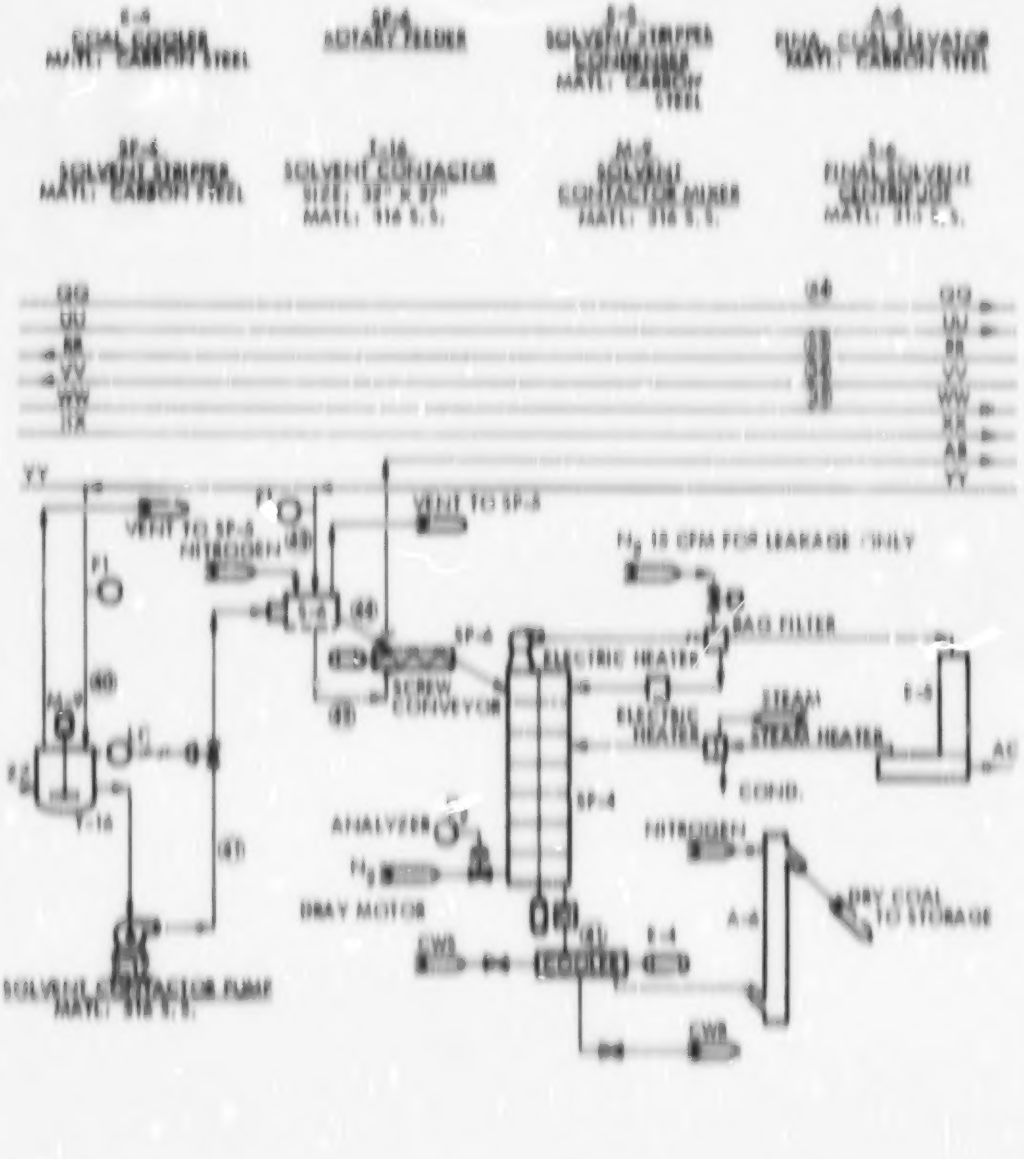
1. THE COAL PULVERIZING SYSTEM IS DESIGNED TO PRODUCE IN THE RANGE 100% MINUS 8 MESH TO 100% MINUS 100 MESH.
2. NORMAL OPERATING CONDITIONS FOR THE FOLLOWING EQUIPMENT ARE 50 PSIG AT 250 °F AND ALTERNATE CONDITIONS ARE 100 PSIG AT 250 °F.
   - A. P-1 SLURRY FEED PUMP
   - B. R-1 PRIMARY REACTOR
   - C. V-1 COMPRESSOR SUCTION KNOCK OUT DRUM
   - D. P-2 A THRU 7 (R-1 REACTOR CIRCULATING PUMPS)
   - E. K-1 OXYGEN RECYCLE COMPRESSOR
   - F. M-2A THRU 7 SLURRY MIXERS
   - G. R-2 SECONDARY REACTOR
   - H. P-3 REACTOR DISCHARGE PUMP
   - I. M-10 & 11 SECONDARY REACTOR MIXERS

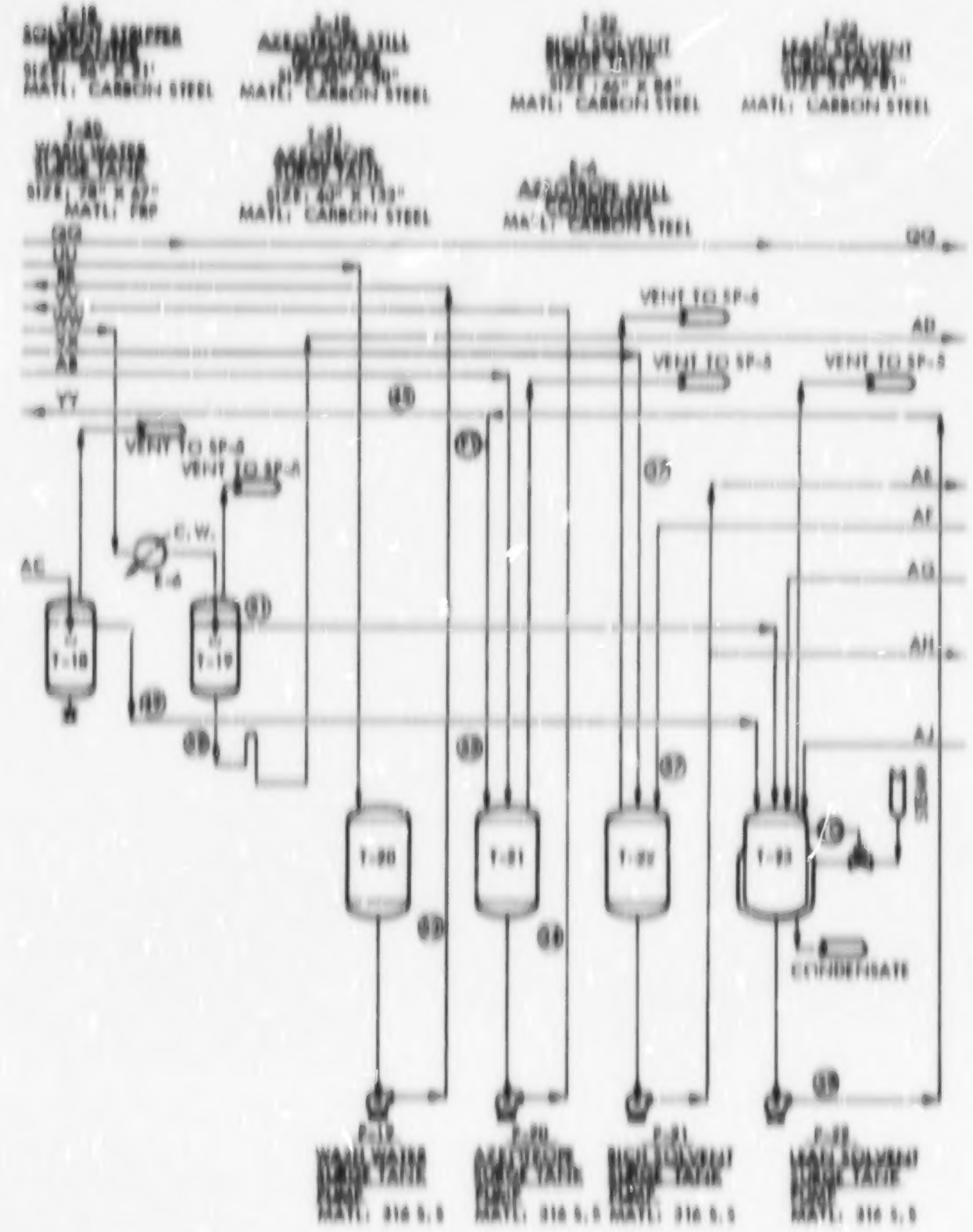
TRW
ONE SPACE PARK · REDONDO BEACH, CALIFORNIA
TRW (MEYERS) COAL DESULFURIZATION
PILOT PLANT
PROCESS FLOW DIAGRAM
0087-AF-1

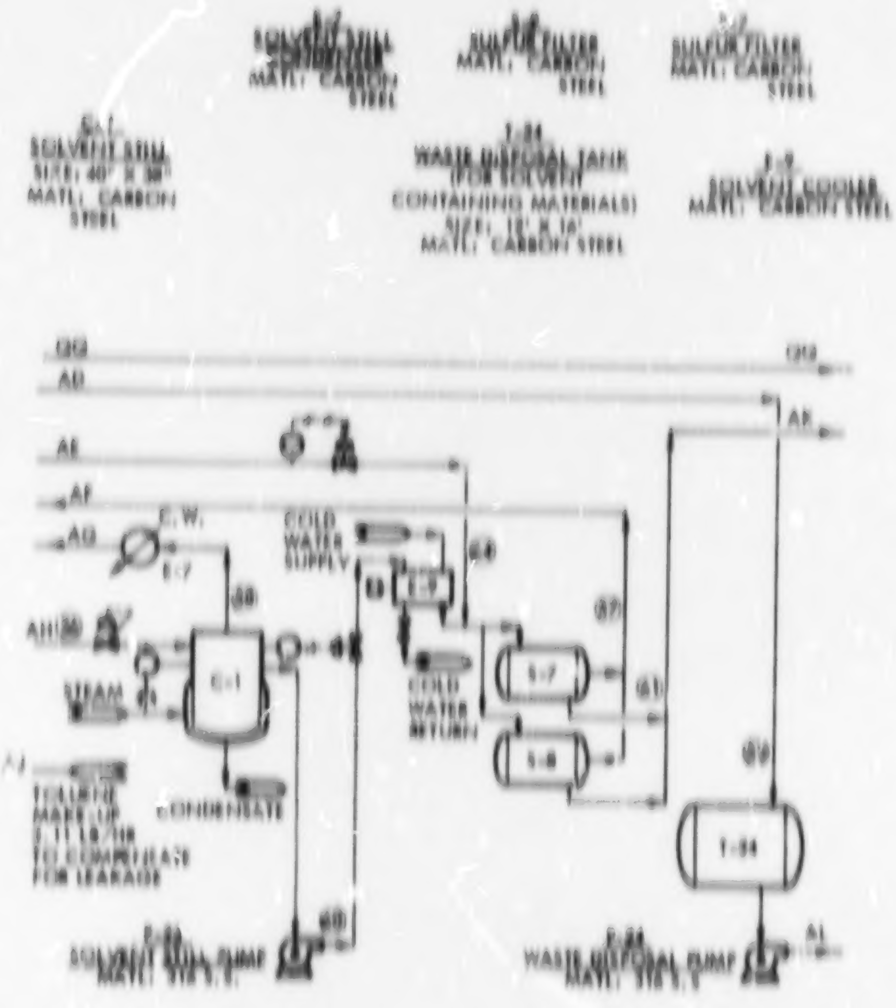

WASH WATER FILTER VACUUM PUMP
SCFM AIR (SATURATED)
MATL: CARBON STEEL
M-7
FINAL WASH WATER CONTACTOR MIXER
MATL: 316 S.S.
T-12
FINAL WASH WATER CONTACTOR
MATL: FRP
T-11
WASH WATER CONTACTOR
MATL: FRP
M-6
WASH WATER CONTACTOR MIXER
MATL: 316 S.S.
S-3
WASH WATER
MATL: 316 S.S.
V-4
FILTRATE RECEIVER
MATL: 316 S.S.
V-5
WASH WATER RECEIVER
MATL: 316 S.
WASH WATER FILTER BAROMETRIC CONDENSER
MATL:
CARBON STEEL
TO FILTER S-2
FROM FILTER S-2
WASH WATER
STEAM
COOLING WATER
COOLING WATER TO COOLING SYSTEM
COOLING WATER TO COOLING SYSTEM
WASH WATER CONTACTOR PUMP
MATL: 316 S.S.
FILTRATE PUMP
MATL: 316 S.S.
WASH WATER PUMP
MATL: 316 S.S.
FINAL WASH WATER CONTACTOR PUMP
MATL: 316 S.S.

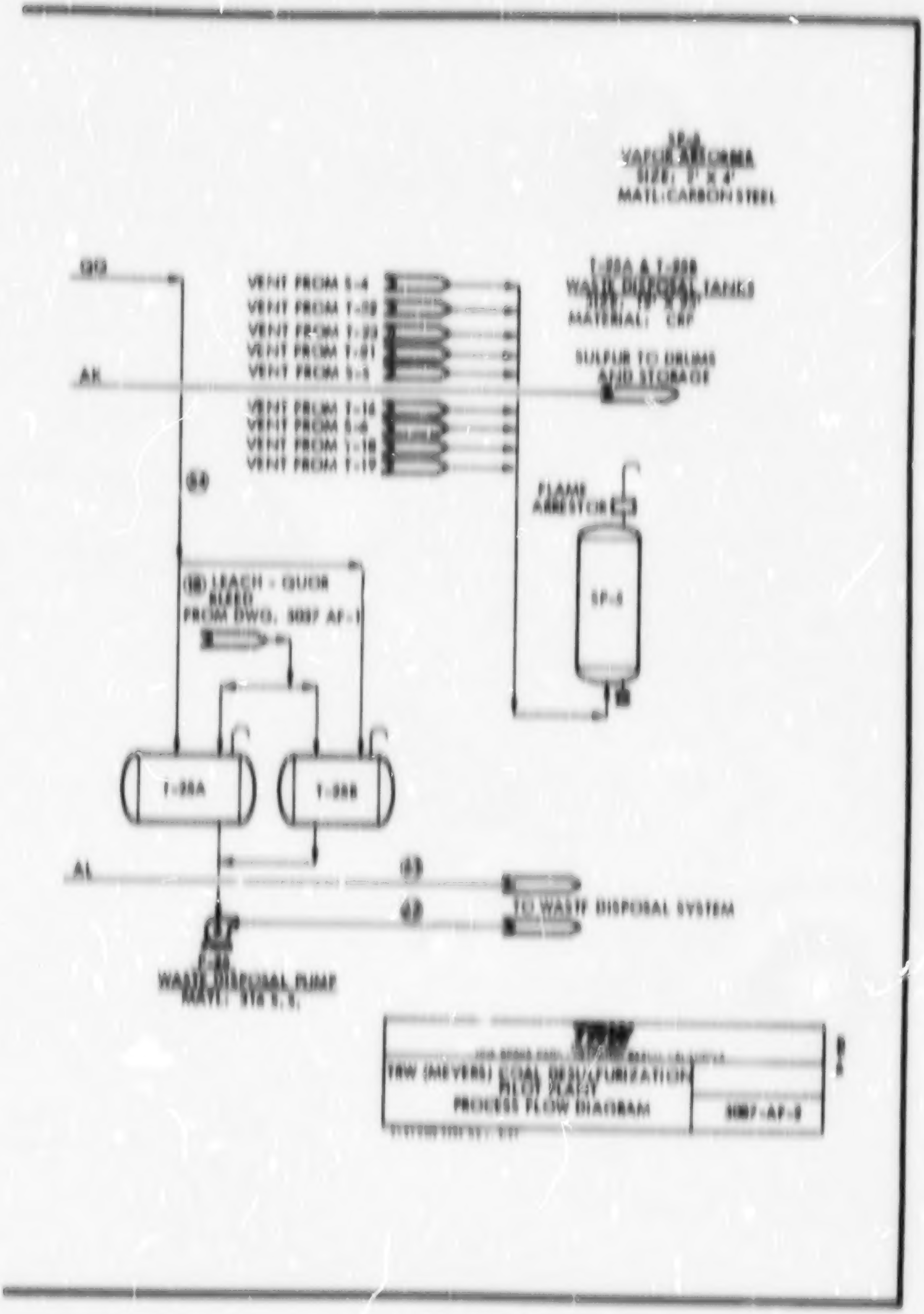


SP-7
ROTARY FEEDER
MAT'L: CARBON STEEL
MAT'L: 316 S.S.
FINAL WASH WATER FILTER
SIZE: 33" X 5' DRUM
FINAL WASH WATER VACUUM PUMP
SCFM AIR SATURATED
FINAL WASH WATER FILTER RECEIVER
SIZE: 42" X 48"
QQ
UU
RR
VV
WW
XX
YY
ZZ
SS
TT
VENT TO SP-6
COOLING WATER
COOLING WATER TO COOLING SYSTEM
NITROGEN
STEAM
FI
LIC
TRC
V-4
T-14
T-15
33
34
35
36
37
38
CONDENSATE


E-4
COAL COOLER
MAT'L: CARBON STEEL
SF-4
ROTARY FEEDER
E-5
SOLVENT STRIPPER CONDENSER
MAT'L: CARBON STEEL
A-4
FINAL COAL ELEVATOR
MAT'L: CARBON STEEL
SP-4
SOLVENT STRIPPER
MAT'L: CARBON STEEL
T-16
SOLVENT CONTACTOR
SIZE: 32" X 97"
MAT'L: 316 S.S.
M-9
SOLVENT CONTACTOR MIXER
MAT'L: 316 S.S.
S-6
FINAL SOLVENT CENTRIFUGE
MAT'L: 316 S.S.
YY
VENT TO SP-5
NITROGEN
VENT TO SP-5
H2 15 CFM FOR LEAKAGE ONLY
BAG FILTER
SF-4
ELECTRIC HEATER
SCREW CONVEYOR
ELECTRIC HEATER
STEAM
STEAM HEATER
E-5
AC
COND.
SP-4
T-16
ANALYZER
NITROGEN
DRY COAL TO STORAGE
A-4
CWS
E-4
CWR
SOLVENT CONTACTOR PUMP
MAT'L: 316 S.S.

MAT'L: CARBON STEEL
MAT'L: CARBON STEEL
MAT'L: CARBON STEEL
MAT'L: CARBON STEEL
MAT'L: FRP
MAT'L: CARBON STEEL
MAT'L: CARBON STEEL
VENT TO SP-5
VENT TO SP-5
VENT TO SP-5
VENT TO SP-5
VENT TO SP-5
AD
AE
AF
AG
AH
AJ
AC
YY
C.W.
E-4
T-18
T-19
T-20
T-21
T-22
T-23
CONDENSATE
MAT'L: 316 S.S
MAT'L: 316 S.S
MAT'L: 316 S.S
MAT'L: 316 S.S


SOLVENT STILL
MAT'L: CARBON STEEL
SULFUR FILTER
MAT'L: CARBON STEEL
SULFUR FILTER
MAT'L: CARBON STEEL
SOLVENT STILL
MAT'L: CARBON STEEL
WASTE DISPOSAL TANK
CONTAINING MATERIALS)
MAT'L: CARBON STEEL
SOLVENT COOLER
MAT'L: CARBON STEEL
AD
AE
AF
AG
AH
AJ
AK
AL
C.W.
E-7
COLD WATER SUPPLY
COLD WATER RETURN
STEAM
E-1
T-7
T-8
T-34
CONDENSATE
TOLUENE MAKE-UP
TO COMPENSATE FOR LEAKAGE
SOLVENT STILL PUMP
WASTE DISPOSAL PUMP


SF-6
VAPOR ABSORBER
SIZE: 1' X 4'
MATL: CARBON STEEL
T-25A & T-25B
WASTE DISPOSAL TANKS
MATERIAL: CRP
VENT FROM S-4
VENT FROM T-22
VENT FROM T-23
VENT FROM T-21
VENT FROM S-5
VENT FROM T-16
VENT FROM S-6
VENT FROM T-18
VENT FROM T-19
AK
SULFUR TO DRUMS
AND STORAGE
64
FLAME
ARRESTOR
SF-6
LEACH - QUOR
BLEED
FROM DWG. 5087 AF-1
T-25A
T-25B
AL
43
42
TO WASTE DISPOSAL SYSTEM
P-26
WASTE DISPOSAL PUMP
MATL: 316 S.S.
TRW
ONE SPACE PARK, REDONDO BEACH, CALIFORNIA
TRW (MEYERS) COAL DESULFURIZATION
PILOT PLANT
PROCESS FLOW DIAGRAM
5087-AF-3

APPENDIX B

PROCESS MASS BALANCE
COMPUTER PROGRAM

APPENDIX B

PROCESS MASS BALANCE COMPUTER PROGRAM

A Fortran computer code prepared for the TRW time-sharing computer is reproduced in the following nine pages. The design basis, upon which the mass balance is based, was discussed in detail in Section 3.0 of the report. The internal data and input data are as follows:

ZIN and Z are the molecular weight of the 10 components.

AA are the names of the 10 components.

X(I,J) is the mole matrix (I=component, J=stream number).

W(I,J) is the identical weight matrix.

XT and WT are the total moles and weight for each stream.

P is the reactor pressure (psig).

Y is the recycle Fe^{+3}/Fe total ratio.

SOL is the solution/coal weight ratio in the mixer feed.

FE is the weight percent iron in the mixer feed.

S is the weight percent pyritic sulfur in the coal.

RX(1) is the percent of pyrite reacted at mixer exit.

(2) is the percent of pyrite reacted at exit of R-1.

(3) is the percent of pyrite reacted at exit of R-2.

VENT is the weight fraction oxygen in dry vent gas.

W(7,1) is moisture on feed coal per 1000 lbs of dry coal.

(7,3) is steam required to heat mixer charge.

(7,11) is flash steam from R-1 exit.

(7,46) is steam required to strip solvent on dryer.

NEW is zero for current flow sheet (1 for a proposed change).

POREW is pore water/dry coal ratio.

SURFW is surface water/dry coal ratio.

PORET is pore toluene/dry coal ratio.

SURFT is surface toluene/dry coal ratio.

RECYC is oxygen fed to R-1/oxygen consumed ratio.

P1 is partial pressure of steam in R-1 gas.

P2 is partial pressure of steam in vent gas.

EXCESS is actual toluene vaporize/theoretical azeotrope toluene.

HOLD is the gpm/ft^2 in the sulfur filter.

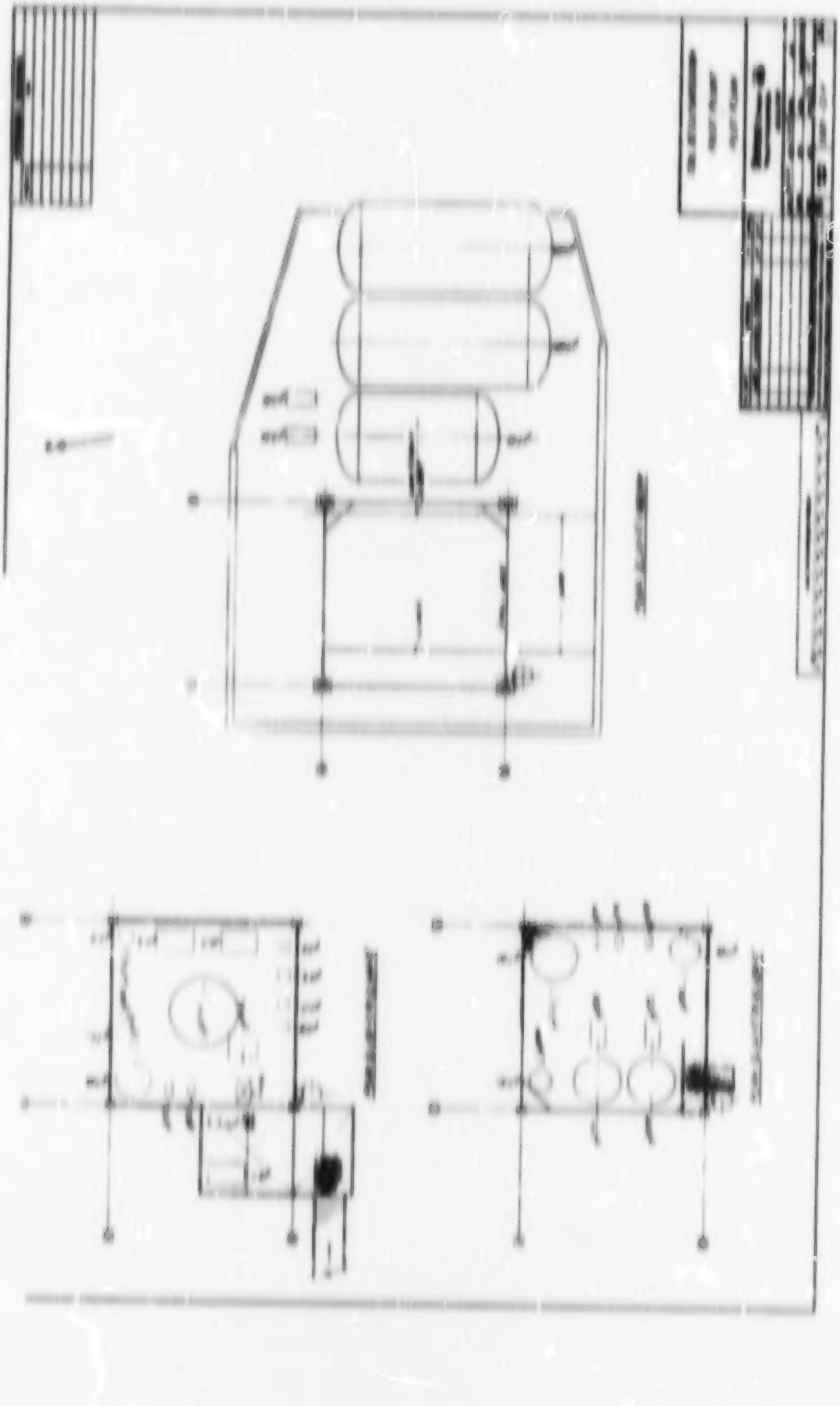
```
      PROGRAM MASSBAL(INPUT,OUTPUT,TAPE1=INPUT,TAPE2,TAPE3)
C     WRITTEN 09/17/76, UPDATED 12/17/76
      COMMON W(10,64),X(10,64),Z(10),Q(7),XT(64)
      DIMENSION RX(3),WT(64),NN(4),AA(10),ZIN(10)
      NAMELIST/WWW/P,T,SOL,FE,S,RX,VENT,W,NEW,
     *POREW,SURFW,PORET,SURFT,REDYO,P1,P2,EXCESS,
     *HOLD
      DATA(ZIN(I),I=1,10)/12.,119.982,32.066,151.915,151.949,
     *98.082,18.016,92.141,32.,35./,
     *(AA(I),I=1,10)/10HCOAL      ,10HPYRITE    ,
     *10HSULFUR    ,10HFESO4     ,10HFE2(SO4)1.5,10HH2SO4     ,
     *10HWATER     ,10HTOLUENE   ,10HOXYGEN    ,10HINERT GAS /
      REWIND 2
      REWIND 3
      DO 96 I=1,10
   96 Z(I)=ZIN(I)
      DO 98 I=1,64
      XT(I)=0.
      WT(I)=0.
      DO 98 J=1,10
      W(J,I)=0.
   98 X(J,I)=0.
C     WEIGHT MATRIX (W) AND MOL MATRIX (X) ZEROED
C     ACCEPT NAMELIST INPUT AND START CALCULATIONS
C     BASIS OF CALCULATIONS--1000 LBS DRY COAL
      READ(2,WWW)
      DISPLAY* ENTER CHANGES, THEN $*
      READ(1,WWW)
      FET=SOL*FE/55.85
      X(5,2)=Y*FET
      X(4,2)=FET-X(5,2)
      X(2,1)=SPW/119.98
      W(2,1)=X(2,1)*Z(2)
      W(1,1)=1000.-W(2,1)
      CALL MOLS(1)
      CALL MOLS(2)
      RXP=RX(3)*X(2,1)/100.
      X(3,2)=0.5*X(4,2)+0.5*RXP
      CALL LBS(2)
      DO 100 I=1,3
  100 WT(I)=WTOT(I)
      W(7,2)=1000.*(SOL+1.)-WT(1)-WT(2)-WT(3)
      X(7,2)=W(7,2)/Z(7)
      WT(2)=WT(2)+W(7,2)
      CALL REACT(XX(1))
      Q(1)=0.
      DO 102 I=1,7
  102 X(I,4)=X(I,1)+X(I,2)+X(I,3)+Q(I)
      CALL LBS(4)
      WT(4)=WTOT(4)
C     STREAMS 1 THROUGH 4 ARE COMPLETE
C     BEGIN OXYGEN BALANCE
      OX=RVP*(P.3*V/4.)*Z(9)
      WT(7)=OX/(0.995-(0.005*VENT/(1.-VENT)))
      W(9,7)=0.995*WT(7)
      W(10,7)=WT(7)-W(9,7)
      W(9,9)=W(9,7)-OX
      W(10,9)=W(10,7)
      CALL MOLS(9)
      PSIA=P+14.7
      X(7,9)=P2*(X(9,9)+X(10,9))/(PSIA-P2)
      W(7,9)=X(7,9)*Z(7)
      CALL MOLS(7)
      WT(9)=WTOT(9)
C     STREAMS 7 AND 9 ARE COMPLETE
C     BEGIN OXYGEN LOOP CALCULATIONS
      W(9,5)=RECYC*OX
      W(9,6)=W(9,7)-OX
      W(10,6)=W(9,6)*(1.-VENT)/VENT
      CALL MOLS(6)
      X(7,6)=P1*(X(9,6)+X(10,6))/(PSIA-P1)
      W(7,6)=X(7,6)*Z(7)
      WT(6)=WTOT(6)
      XX=P2*(X(9,6)+X(10,6))/(PSIA-P2)
      XX=X(7,6)-XX
      CALL SUMX(6,-1,8)
      X(7,8)=X(7,9)+XX
      CALL SUMX(7,8,5)
      CALL SAME(2,17)
      X(7,17)=X(7,2)-XX
      CALL LBS(5)
      CALL LBS(8)
      CALL LBS(17)
      WT(5)=WTOT(5)
      WT(8)=WTOT(8)
      WT(17)=WTOT(17)
C     STREAMS 5,6,8,17 ARE COMPLETE
C     BEGIN REACTOR R-1 CALCULATIONS
      CALL SAME(4,10)
      CALL REACT(RX(2)-RX(1))
      DO 104 I=2,7
  104 X(I,10)=X(I,10)+D(I)
      OXM=OX/Z(4)
      X(4,10)=X(4,10)-4.*OXM
      X(5,10)=X(5,10)+5.*OXM
      X(6,10)=X(6,10)-2.*OXM
      X(7,10)=X(7,10)+2.*OXM+X(7,5)-X(7,6)
C     STREAM 10 COMPLETE FOR REACTOR R-1
C     BEGIN REACTOR R-2
      X(7,11)=W(7,11)/Z(7)
      CALL SUMX(10,-11,12)
      CALL SAME(12,13)
      CALL REACT(RX(3)-RX(2))
      DO 106 I=2,7
  106 X(I,13)=X(I,13)+D(I)
      DO 108 I=10,13
      CALL LOST(I)
  108 WT(I)=WTOT(I)
C     STREAMS 11,12,13 ARE COMPLETE
C     BEGIN WASHING AND FILTER SECTION
      SOLID=W(1,13)+W(2,13)+W(3,13)
      WATER=SOLID*(POREW+SURFW)
      F1=(W(7,13)-WATER)/W(7,13)
      DO 110 I=4,7
  110 X(I,19)=X(I,13)*F1
C     UNWASHED CAKE TEMPORARILY IS STREAM 53
      CALL SUMX(13,-19,53)
      F1=1.4*WATER/(1.4*WATER+1.5*SOLID)
      F2=SURFW/(POREW+SURFW)
      F3=0.4/1.4
      F4=WATER/(2.*SOLID)
      F5=(1.-F2)*F4
C     BEGIN ITERATION OF WASH LOOP
      DO 118 I=1,100
      DO 112 J=4,8
  112 X(J,22)=X(J,32)*F1
```

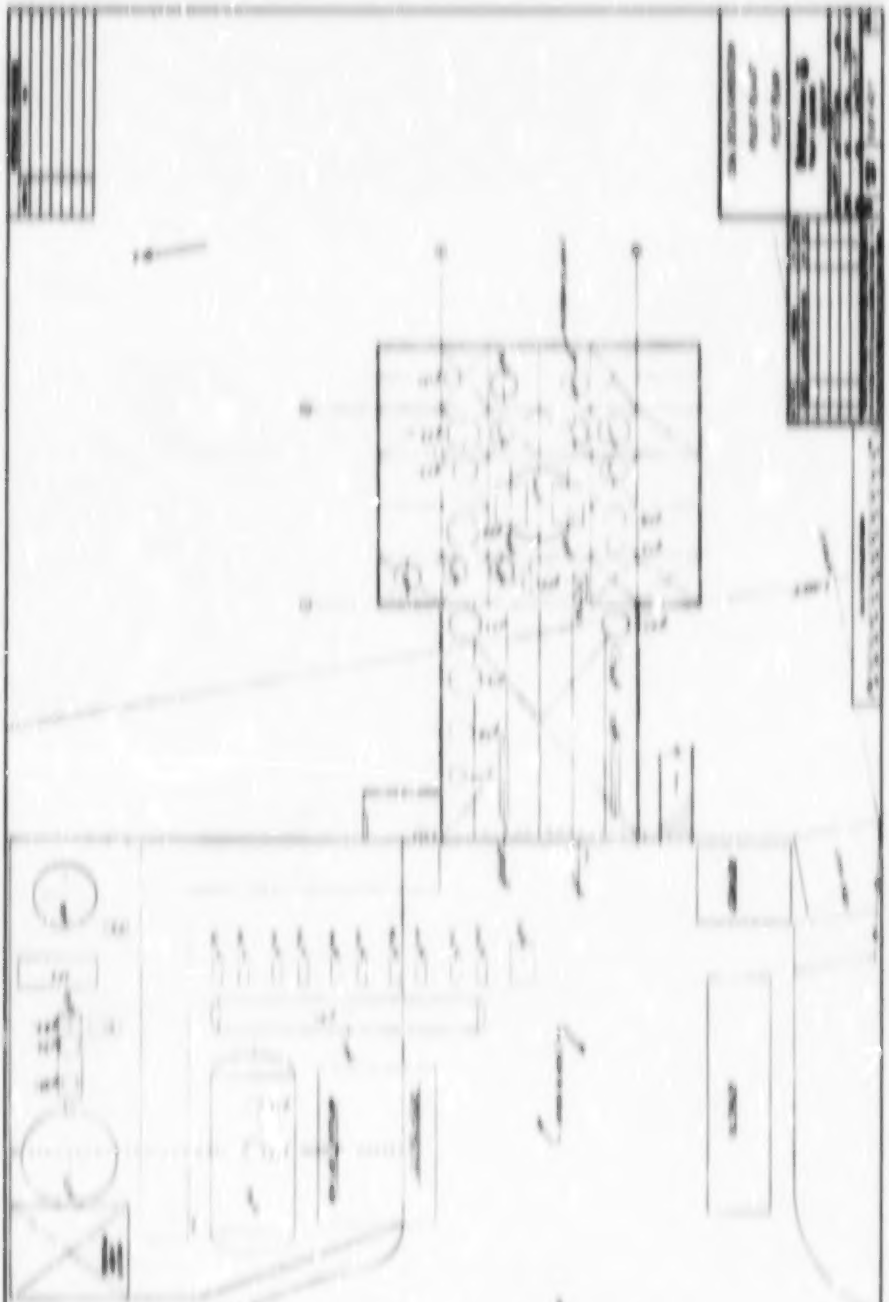
```
      CALL SUMS(32,-22,23)
      DO 114 J=4,6
  114 X(J,30)=X(J,53)*F2+X(J,22)*F3
      CALL SUMX(53,-20,21)
      CALL SUMS(21,22,21)
      CALL SUMS(21,23,24)
      DO 116 J=4,6
      X(J,28)=X(J,24)*F5
  116 X(J,34)=X(J,28)*F5
      IF(ABS(X(5,28)*(1-F5)-X(5,32)).LT.1.0E-7)GO TO 120
      CALL SUMS(28,-33,32)
      IF(I.EQ.99)DISPLAY" SO4 LOOP NOT CONVERGED"
  118 CONTINUE
C     FERRIC SULFATE IS BALANCED TO 0.0000001 MOLE
C     CALCULATE STREAMS IN WASH LOOP
  120 X(7,20)=1.4*WATER/Z(7)
      X(7,22)=X(7,20)
      X(7,26)=X(7,20)
      X(7,21)=WATER/Z(7)
      X(7,23)=1.5*SOLID/Z(7)
      CALL SUMX(21,23,24)
      X(7,20)=X(7,21)
      X(7,27)=X(7,20)
      X(7,31)=X(7,20)
      X(7,29)=X(7,23)
      X(7,25)=X(7,23)
      DO 122 I=4,6
      X(I,25)=X(I,24)*(1.-F4)
      X(I,20)=(X(I,24)-X(I,25))*F2
  122 X(I-3,28)=X(I-3,24)
      CALL SUMX(28,29,30)
      CALL SUMX(29,20,33)
      CALL SUMX(22,23,32)
      CALL SAME(32,33)
      DO 124 I=1,7
  124 X(I,30)=X(I,13)
      X(7,33)=X(7,21)
      X(7,40)=X(7,27)+X(7,29)+X(7,31)
C     COMPLETED WASH LOOP MOLE BALANCE
C     CALCULATE RECYCLE AND BLEED STREAMS
      CALL SUMX(17,-15,16)
      F1=AMP-X(4,34)-X(5,34)-X(4,32)-X(5,32)
      F2=F1/(X(4,20)+X(5,20))
      DO 126 I=4,7
  126 X(I,18)=X(I,20)*F2
      CALL SJMX(20,-18,15)
      CALL SJMX(16,-15,19)
      CALL SJMX(18,54,62)
C     COMPLETED RECYCLE, MAKEUP, AND BLEED CALCULATION
C     START TOLUENE EXTRACTION CALCULATIONS
      SOLID=W(1,13)+W(2,13)
      TOL=(PORE1+SURF1)*SOLID
      W(3,33)=W(3,13)
      W(7,35)=X(7,33)*Z(7)
      W(7,52)=W(7,35)
      W(8,35)=X(7,33)*44.4*EXCESS*Z(8)/55.6
      W(8,34)=2.*SOLID
      W(8,38)=2.*TOL
      W(8,39)=TOL
      W(8,40)=(2.*SOLID)-TOL
      W(8,41)=W(8,38)
      W(8,43)=W(8,38)
      W(8,44)=W(8,39)
      W(8,45)=W(8,44)
      W(8,51)=W(8,35)
      W(8,37)=2.*SOLID+TOL
      W(8,50)=W(8,37)
      W(8,59)=W(8,45)+W(8,51)+W(8,50)
      W(8,55)=W(8,59)-W(8,18)-W(8,40)-W(8,43)
      W(8,42)=W(8,37)
      W(8,36)=W(8,42)+W(8,55)
C     TOLUENE WEIGHT BALANCE COMPLETE EXCEPT STILL
C     BEGIN SULFUR BALANCE BY ITERATING RECYCLE LOOP
      F1=(W(8,38)-W(8,39))/W(8,38)
      F2=0.5*SURF1/(SURF1+PORE1)
      F3=F1+F2-F1*F2
      F4=1.-F3
      DO 128 I=1,100
      W(3,36)=W(3,33)+W(3,34)
      W(3,39)=F4*W(3,36)
      W(3,42)=F3*W(3,39)
      IF(ABS(W(3,42)-W(3,34)).LT.1.0E-4)GO TO 130
      IF(I.EQ.99)DISPLAY" SULFUR LOOP NOT CONVERGED"
  128 W(3,34)=W(3,42)
```

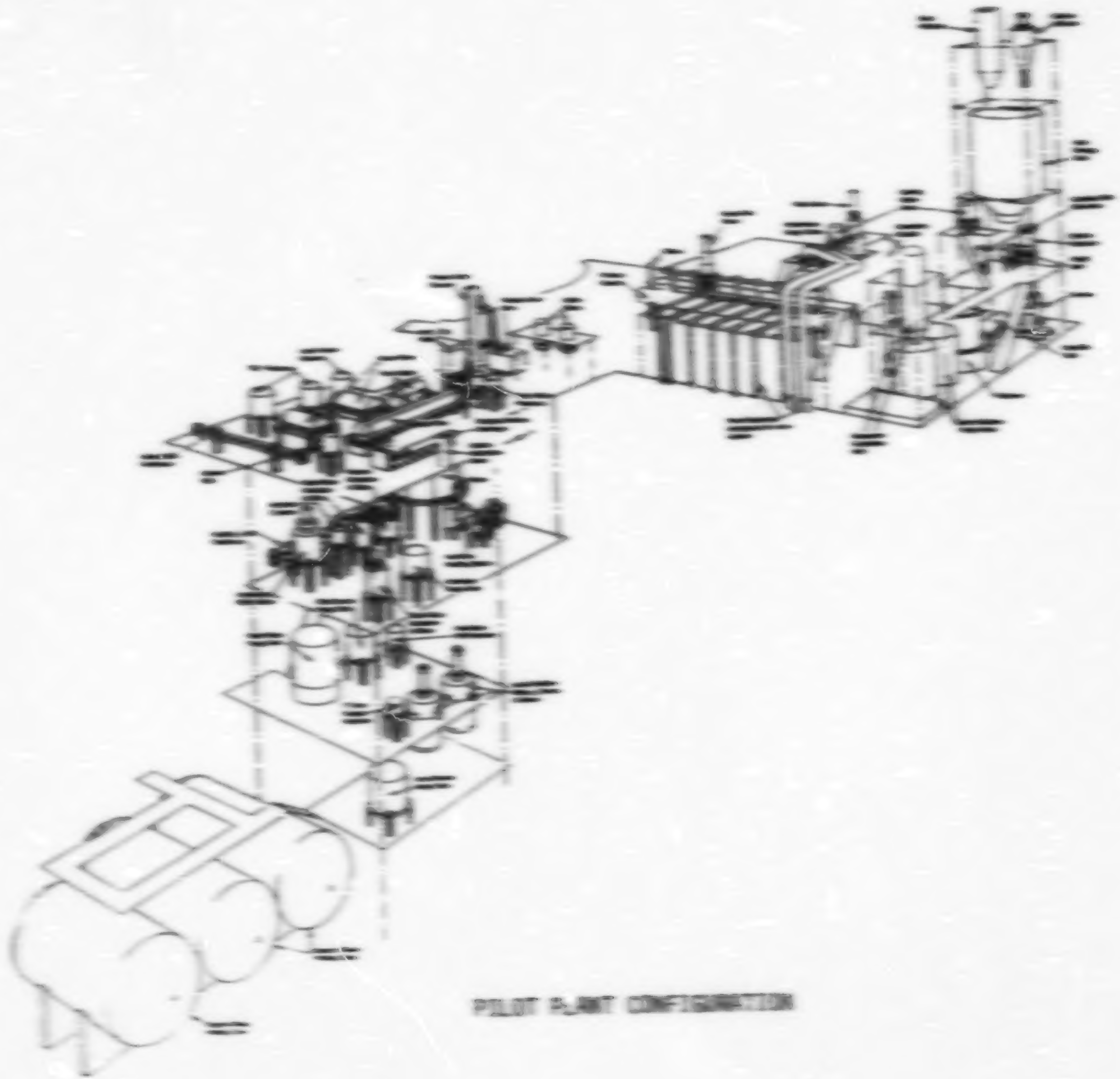
```
C      SULFUR LOOP CONVERGED TO 0.0001 LB
  130 W(3,34)=W(3,42)
      W(3,36)=W(3,33)+W(3,34)
      W(3,37)=W(3,36)*F3
      W(3,39)=W(3,36)-W(3,37)
      W(3,41)=W(3,39)
      W(3,44)=W(3,41)-W(3,42)
      W(1,57)=W(3,44)
C      BEGIN STILL CALCULATIONS--STILL BOTTOM 10 PCT SULFUR
C      FILTER FEED SATURATED AT 99F, OR 2.7 PCT SULFUR
C      STREAM 64 TO CAKE HOLDER (1.0 GPM/FT2=28800 LB/HR)
      IF(NEW.NE.1)GO TO 134
C      TWO OPTIONS: 132 FOR NEW, 134 FOR OLD
C      BASED ON NEW FLOWSHEET--DIRECT RECYCLE OF HOLD LIQUID
  132 F1=0.1
      F2=0.027
      F3=(1.-F2)/(1.-F1)
      W(3,60)=W(3,37)*F3/(F3-(F2/F1))
      WT(60)=W(3,60)/F1
      W(8,60)=WT(60)-W(3,60)
      W(3,61)=W(3,37)
      W(3,56)=W(3,60)
      W(8,56)=W(8,58)+W(8,60)
      WT(57)=28800.*HOLD
      W(3,57)=F2*WT(57)
      W(8,57)=WT(57)-W(3,57)
      W(8,64)=W(8,57)-W(8,58)
      W(3,64)=W(3,57)+W(3,61)-W(3,60)
      GO TO 136
C      DIRECT RECYCLE NEW FLOWSHEET OPTION COMPLETE
C      BASED ON OLD FLOWSHEET--RECYCLE THROUGH T-22
  134 F1=0.1
      F2=0.027
      WT(57)=28800.*HOLD
      W(3,57)=F2*WT(57)
      W(8,57)=WT(57)-W(3,57)
      F3=W(3,37)+W(3,57)
      F4=W(8,58)+W(8,57)
      F5=F1*W(8,56)/(F1*F4+F1*F3-F3)
      W(3,56)=F5*F3
      W(8,56)=F5*F4
      W(3,60)=W(3,56)
      W(8,60)=W(8,56)-W(8,54)
      W(3,65)=F3-W(3,56)
      W(4,66)=F4-W(4,56)
      W(1,61)=W(1,37)
C     ALL STREAMS THROUGH THE EXTRACTION SECTION ARE DONE
C     THE FINAL SECTION IS BASED ON NITROGEN STRIPPING
C     STREAMS 48 AND 50 WERE ELIMINATED BY N2 STRIPPING
C     STREAMS 49 AND 46 WERE REASSIGNED
  136 W(7,63)=W(7,52)
      W(8,49)=W(5,38)+W(8,40)+W(8,43)
C     COMPLETE WEIGHT AND MOL CALCULATIONS
      DO 138 I=13,33
      CALL LBS(I)
  138 WT(I)=WTOT(I)
      DO 140 I=1,2
      W(I,36)=W(I,33)
      W(I,39)=W(I,33)
      W(I,41)=W(I,33)
      W(I,44)=W(I,33)
  140 W(I,47)=W(I,33)
      DO 142 I=4,8
      W(I,36)=W(I,33)
      W(I,39)=W(I,33)
      W(I,41)=W(I,33)
      W(I,44)=W(I,33)
  142 W(I,47)=W(I,33)
      CALL LBS(46)
      CALL LBS(53)
      CALL LBS(59)
      CALL LBS(62)
      DO 144 I=36,64
      CALL MOLS(I)
  144 WT(I)=WTOT(I)
C     CALCULATIONS COMPLETE, BEGIN OUTPUT
      DO 206 I=1,8
      NF=8*I
      N=NF-7
      NN(1)=N
      DO 200 J=1,7
  200 NN(J+1)=NN(J)+1
      WRITE(6,1000)I,(NN(K),K=1,8)
      DO 202 J=1,10
      WRITE(3,1002)AA(J),(X(J,K),K=N,NF)
  202 CONTINUE
      WRITE(3,1004)
      DO 204 J=1,10
      WRITE(3,1006)AA(J),(W(J,K),K=N,NF)
  204 CONTINUE
      WRITE(3,1008)(WT(K),K=N,NF)
  206 CONTINUE
 1000 FORMAT(*1*////33X*PILOT PLANT MASS BALANCE*
     +33X*PAGE *I1* OF 8*//*  STREAM NO.*I3,
     *8I12//45X*FLOW RATE, LB-MOL/HR*)
 1002 FORMAT(2X,A10,8F12.4)
 1004 FORMAT(/,45X,*FLOW RATE, LB/HR*)
 1006 FORMAT(2X,A10,8F12.2)
 1008 FORMAT(/*  TOTAL WT.*8F12.2)
      STOP
      END
      SUBROUTINE MOLS(N)
      COMMON W(10,64),X(10,64),Z(10),D(7)
      DO 10 I=1,10
   10 X(I,N)=W(I,N)/Z(I)
      RETURN
      END
      SUBROUTINE LBS(N)
      COMMON W(10,64),X(10,64),Z(10),D(7)
      DO 20 I=1,10
   20 W(I,N)=X(I,N)*Z(I)
      RETURN
      END
      SUBROUTINE REACT(PCT)
      COMMON W(10,64),X(10,64),Z(10),D(7)
      D(2)=-(PCT/100.*X(2,1))
      D(3)=3.2*D(2)
      D(7)=4.8*D(2)
      D(6)=-(D(7))
      D(4)=-(10.2*D(2))
      D(1)=-(8.8*D(2))
      RETURN
      END
      SUBROUTINE SUMX(I,J,K)
      COMMON W(10,64),X(10,64),Z(10),D(7),XT(64)
      XT(K)=0.
      M=1
      IF(J.LT.0)M=-1
      J=J*M
      DO 30 L=1,10
      X(L,K)=X(L,I)+M*X(L,J)
   30 XT(K)=XT(K)+X(L,K)
      J=J*M
      RETURN
      END
      SUBROUTINE SUM3(I,J,K)
      COMMON W(10,64),X(10,64),Z(10),D(7)
      M=1
      IF(J.LT.0)M=-1
      J=J*M
      DO 40 L=1,6
   40 X(L,K)=X(L,I)+M*X(L,J)
      J=J*M
      RETURN
      END
      SUBROUTINE SAME(I,J)
      COMMON W(10,64),X(10,64),Z(10),D(7)
      DO 50 K=1,10
   50 X(K,J)=X(K,I)
      RETURN
      END
      FUNCTION WTOT(N)
      COMMON W(10,64),X(10,64),Z(10),D(7)
      T=0.
      DO 15 I=1,10
   15 T=T+W(I,N)
      WTOT=T
      RETURN
      END
```

APPENDIX C

PILOT PLANT PLOT PLAN
AND CONFIGURATION







PILOT PLANT CONFIGURATION

APPENDIX D

COMPLETE PILOT PLANT EQUIPMENT LIST

COMPLETE EQUIPMENT LIST

[illegible]

COMPLETE EQUIPMENT LIST (CONT'D)

COMPLETE EQUIPMENT LIST (CONT'D)

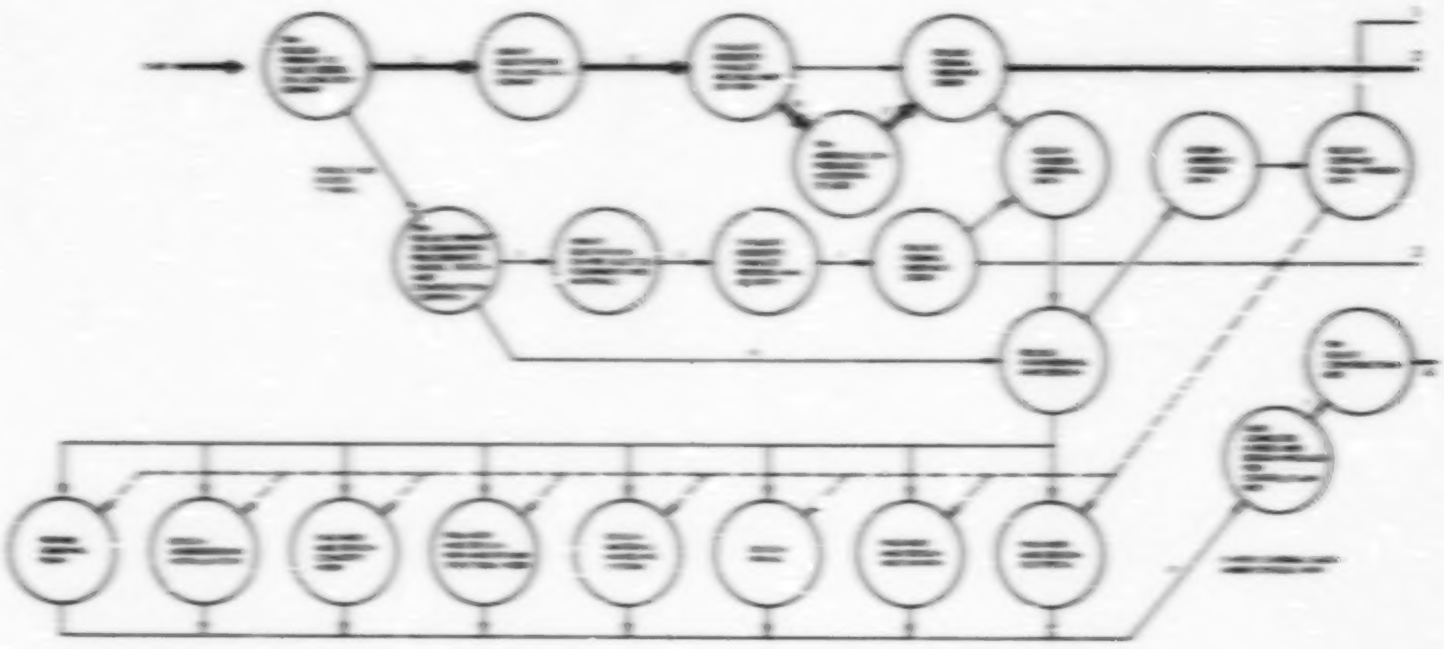
APPENDIX E

CRITICAL PATH DIAGRAM

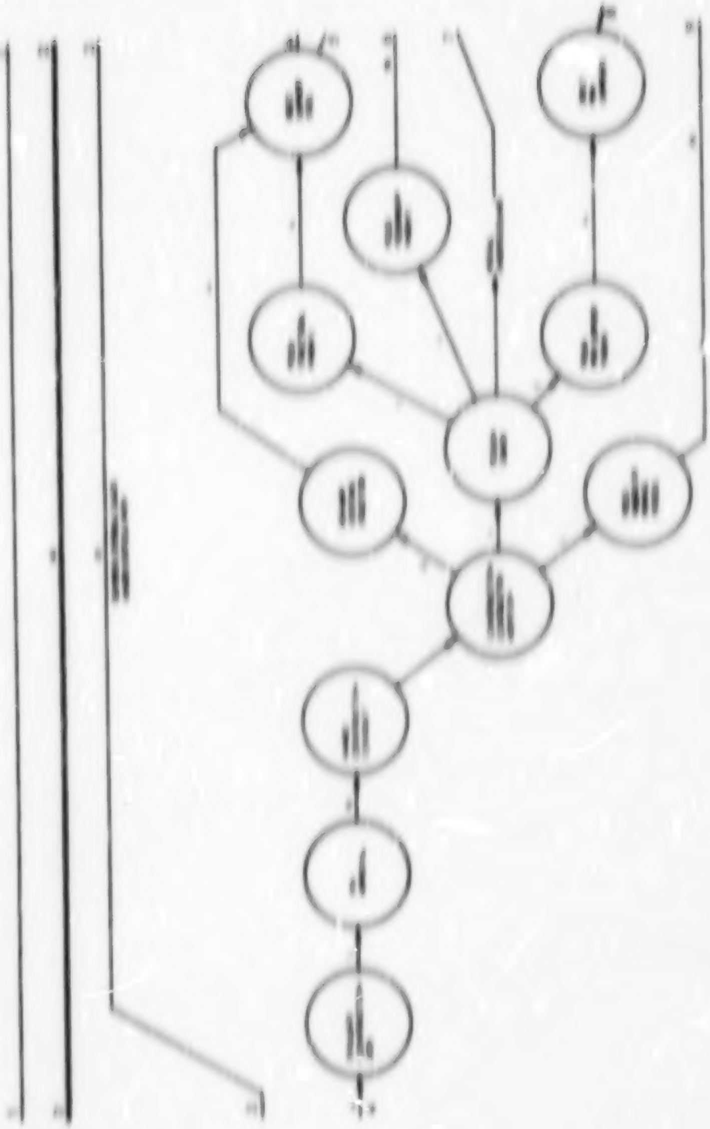
APPENDIX E

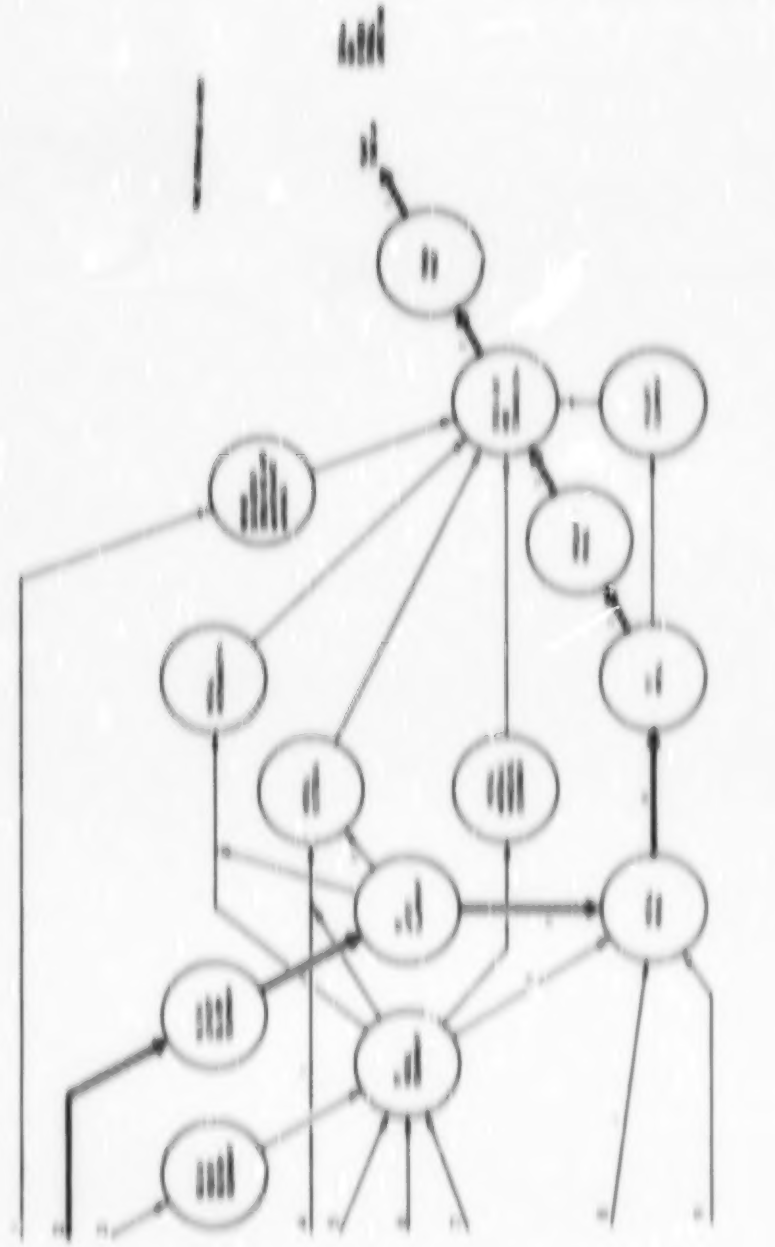
CRITICAL PATH DIAGRAM

A critical path diagram indicating the time phase sequence of pilot plant construction activities is presented on the following page. The critical path, indicated by the dark lines, requires 87 weeks. Of the 87 weeks, there is one pacing item, namely the delivery of the long lead time equipment. This time requirement (65 weeks) is dictated by the quoted delivery schedule for reactors R-1 and R-2. Were the procurement activitites associated with these two items of equipment started prior to the initiation of the presented critical path, a maximum of up to 13 weeks of scheduled time could be saved. The resultant construction schedule would then require 74 weeks.



CRITICAL PATH SCHEDULE FOR PILOT PLANT CONSTRUCTION





APPENDIX F

REACTOR TEST UNIT FLOW DIAGRAM

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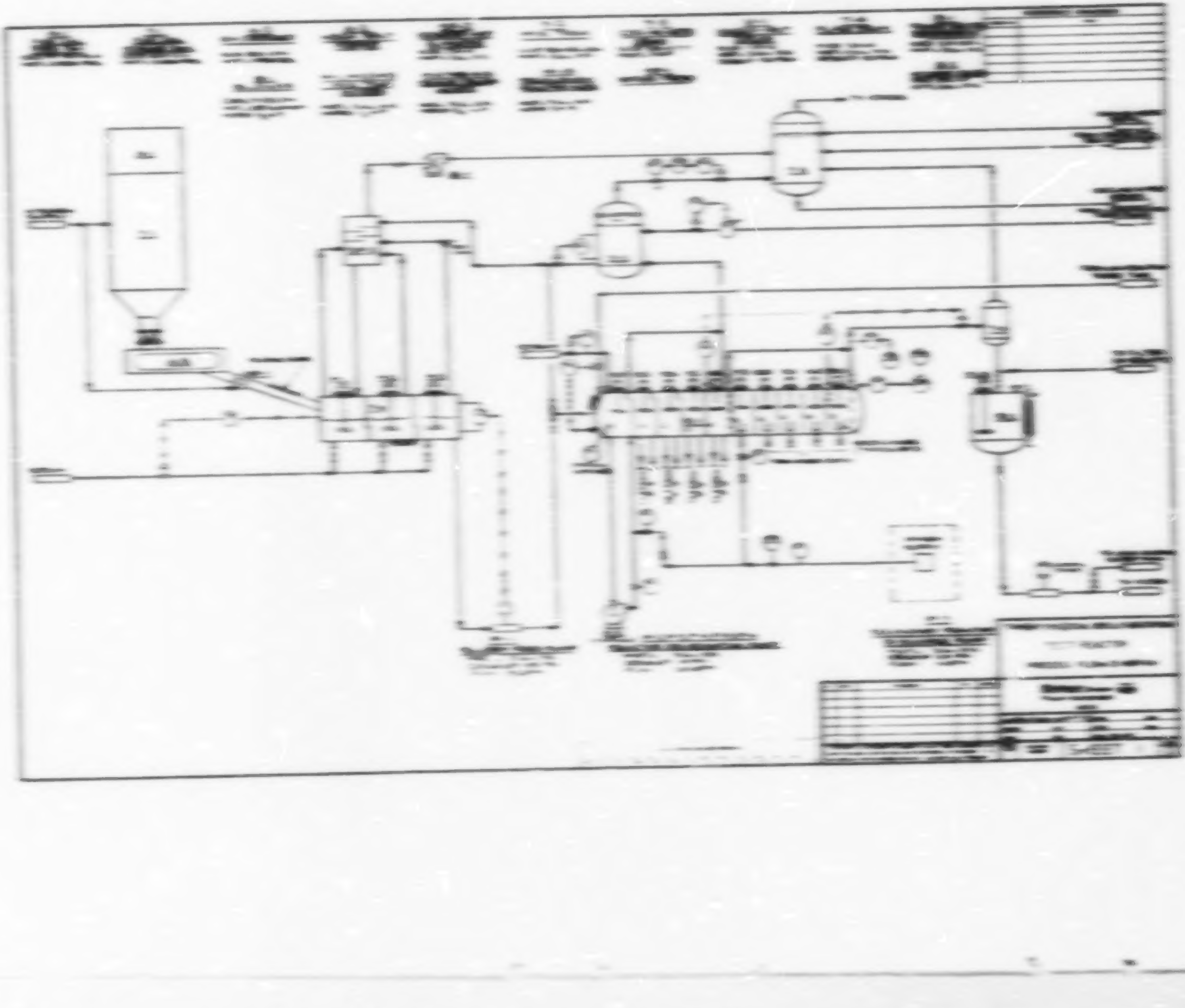

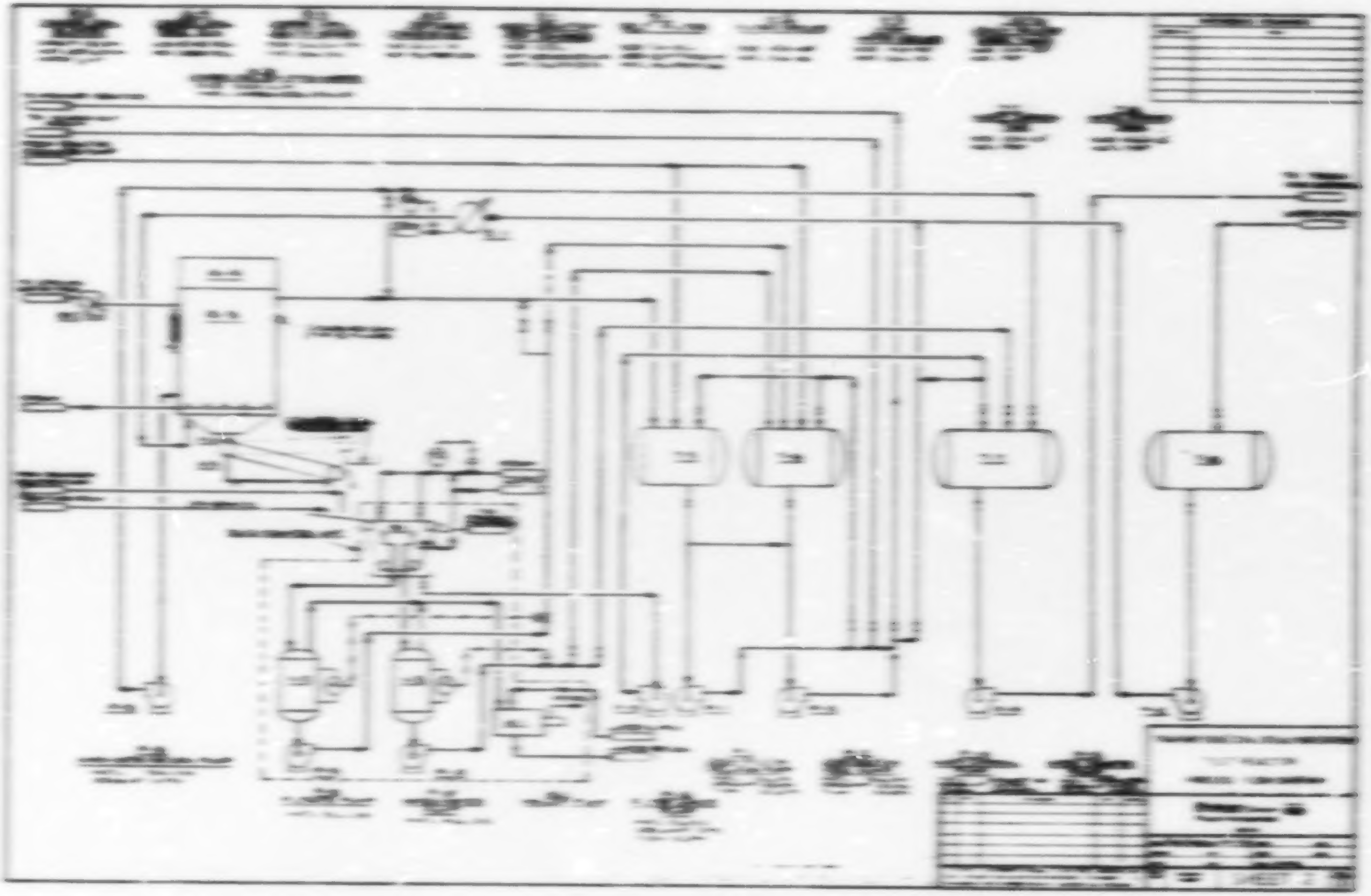

APPENDIX G

REACTOR TEST UNIT PLOT PLAN AND CONFIGURATION

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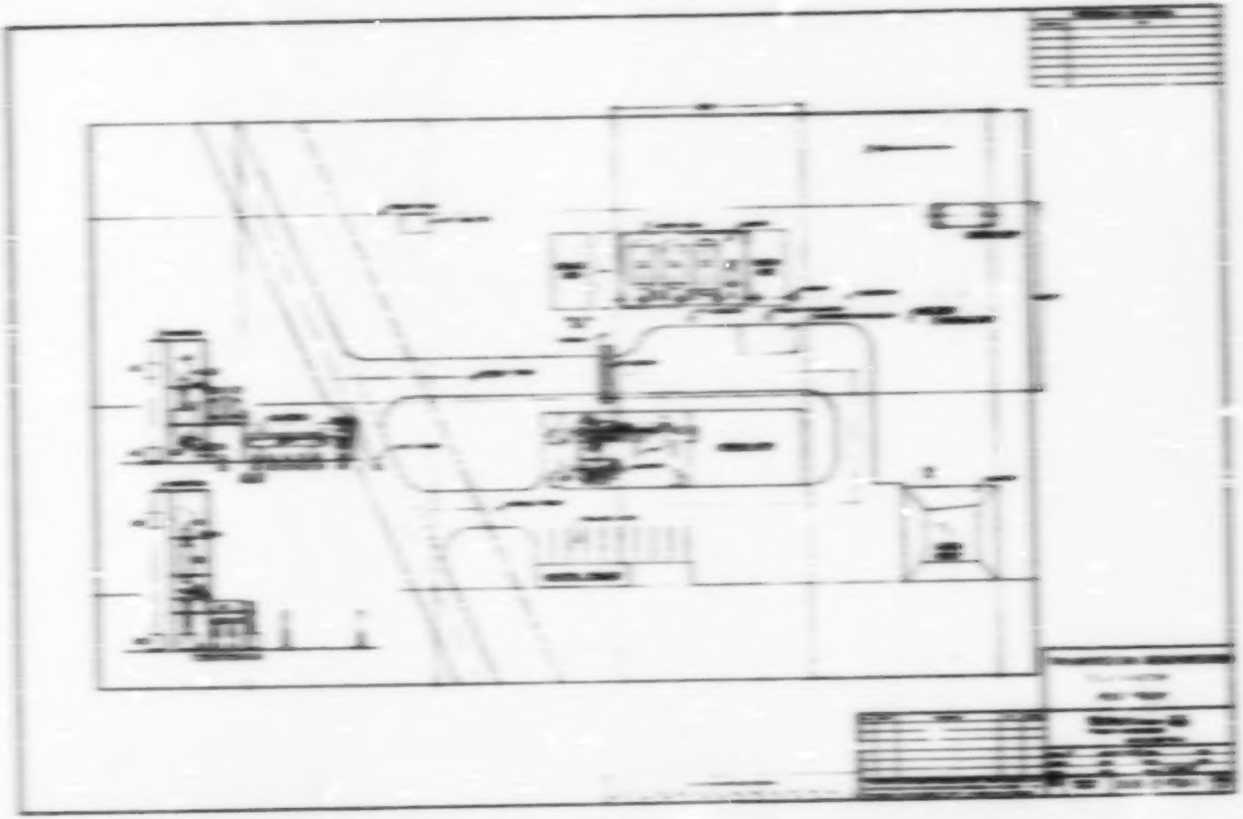
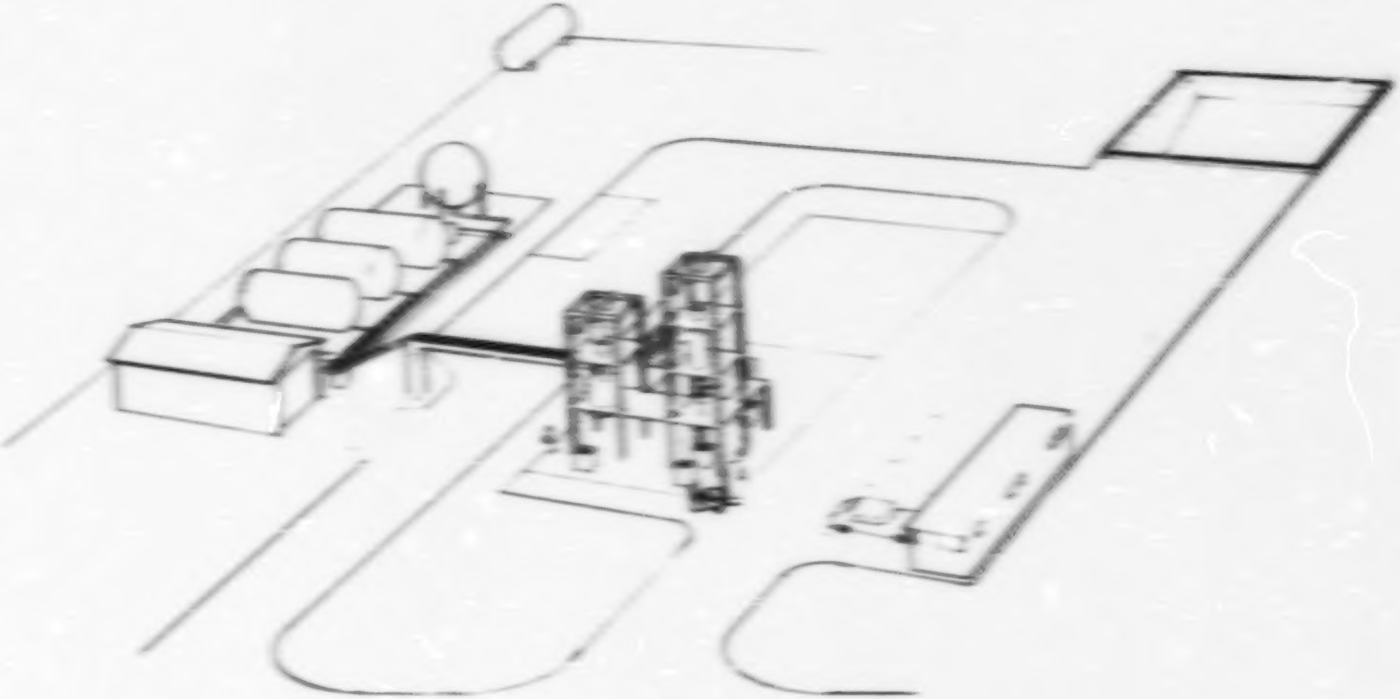


TEST REACTOR PLOT PLAN



ARTIST'S RENDITION - REACTOR TEST UNIT

APPENDIX H

REACTOR TEST UNIT EQUIPMENT LIST

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EQUIPMENT NUMBER	SERVICE	VENDOR	EST. HP	DELIVERY WEEKS	REMARKS	QUANTITY
A-2	BIN DISCHARGER	CARMAN	3/4	10-12		1
A-3	WEIGH BELT FEEDER	K-TRON	1/3	10-12	Includes Sample Valve, and Flex. Connection	1
A-5	PORTABLE CONVEYOR	BUCKET ELEVATOR	1	10-12		1
A-6	COARSE COAL BIN DISCHARGER	TOTE	3/4	10-12		1
T-1	FINE COAL STORAGE TK.	C.E. HOWARD		12		1
T-2	MIX TANK	C.E. HOWARD		12		1
T-3	COOLING WATER DRUM	FIBER-DYNE		12-14		1
T-4	FLUSH TANK	FIBER-DYNE		12-14		1
T-5	LEACH SOLUTION SURGE TK.	FIBER-DYNE		12-14		1
T-6	LEACH SOLUTION SURGE TK.	FIBER-DYNE		12-14		1
T-7	WASTE DISPOSAL TANK	FIBER-DYNE				1
T-8	WATER STORAGE TANK	FIBER-DYNE				1
M-1 M-2 M-3	MIX TANK MIXER	LIGHTNIN	1/3	10		3
M-4 thru M-13	PRIMARY REACTOR MIXER	LIGHTNIN	3/4	10		10
M-14	SECONDARY REACTOR MIXER	LIGHTNIN	3	10		1
E1	COARSE COAL HEAT EXCH.	BROWN		10		1
P-1	SLURRY FEED PUMP	HILLS-McCANNA	3	16-18		1
P-2	REACTOR RECIRCULATION	LABOUR	1	28-30		1
P-3 thru P-6	PUMPS	LABOUR	1	28-30		4
P-7	SECONDARY REACTOR DISC. PUMP	HILLS-McCANNA	3	16-18		1

REACTOR TEST UNIT EQUIPMENT LIST

EQUIPMENT NUMBER	SERVICE	VENDOR	EST. HP	DELIVERY WEEKS	REMARKS	QUANTITY
P-8	WASH PUMP	EASTERN	1/2 115/230 1Ø	4-6	316 S.S.(Purchase Spare)	2
P-9	FILTRATE PUMP	AMETEC	2		W/Filter Package	-
P-10	WASH WATER PUMP	AMETEC	2		W/Filter package	-
P-11	LEACH SOLUTION CIRC. PUMP	DEAN	15	30		1
P-12	LEACH SOLUTION FEED PUMP	SUNDYNE	5	28-30		1
P-13	SCREEN WASTE PUMP	EASTERN	1/2 115/230 1Ø	4-6	316 S.S.	1
P-14	WASTE DISPOSAL PUMP	WESTCO PUMP	10	16-18		1
P-15	WATER TRANSFER PUMP	EASTERN	1/2 115/230 1Ø	4-6		1
V-1	VACUUM PUMP	AMETEC	30		W/Filter Package	-
R-1	PRIMARY REACTOR GENERATOR	C.E. HOWARD		15		1
R-2	SECONDARY REACTOR	C.E. HOWARD		16		1
R-3	COARSE COAL REACTOR WASHER	C.E. HOWARD		16		1
V-1	KNOCK-OUT DRUM	C.E. HOWARD		12		1
V-2	FILTRATE RECEIVER	AMETEC			W/Filter Package	-
V-3	WASH WATER RECEIVER	AMETEC			W/Filter Package	-
B-1	BLOWER	SHARPE HEATING & VENT	1/3 110/230			
B-2	COARSE COAL BLOWER	SHARPE HEATING & VENT	1/3 110/230			-

REACTOR TEST UNIT EQUIPMENT LIST (CONT'D)

EQUIPMENT NUMBER	SERVICE	VENDOR	EST. HP	DELIVERY WEEKS	REMARKS	QUANTITY
SP-1	ROTARY FEEDER	SPROUT WALDRON	1/2	12	0.175 CF/REV	1
SP-2	SCRUBBER & MIST ELIMINATOR	C.E. HOWARD		15		1
S-1	VACUUM FILTER	AMETEC	35-40	1st Quarter of 1976		1
NA	CRANE (FOR COARSE & FINE COAL)	TOTE	10			1
NA	TILT MECHANISM FOR FEED BIN FINE COAL	TOTE		12	Including vibrator need 25 CFM of air for unloading.	1
NA	TILT MECHANISM FOR FEED BIN COARSE COAL	TOTE		12	Including vibrator need 25 CFM of air for unloading.	1

REACTOR TEST UNIT EQUIPMENT LIST (CONT'D)

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(Please read Instructions on the reverse before completing)

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16. ABSTRACT The report gives results of a program for design and operational planning of facilities for testing the Meyers Process for chemical removal of pyritic sulfur from coal. Two options were evaluated: a complete pilot plant test of the process at a 0.5-ton per hour scale; and scale-up and testing of only the most critical portion of the process, the reactor and regenerator section (reactor testing unit). The report includes: a summary of background process data; a discussion of the pilot plant design, pilot plant start-up and operational test plans; and the preliminary design, start-up, and test approach for the reactor testing unit. It also includes: process flow diagrams for the complete pilot plant; pilot plant mass balance computer program; pilot plant plot plans and a sketch of the facility; complete pilot plant equipment list; critical path schedule for construction of the pilot plant; preliminary process flow diagrams for the reactor testing unit approach; preliminary reactor test unit plot plans and a sketch of the facility; and reactor test unit equipment list.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Air Pollution	Air Pollution Control	13B
Coal	Stationary Sources	21D
Coal Preparation	Pyritic Sulfur	08I
Desulfurization	Chemical Processes	07A,07D
Pilot Plants	Meyers Process	
Design		

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